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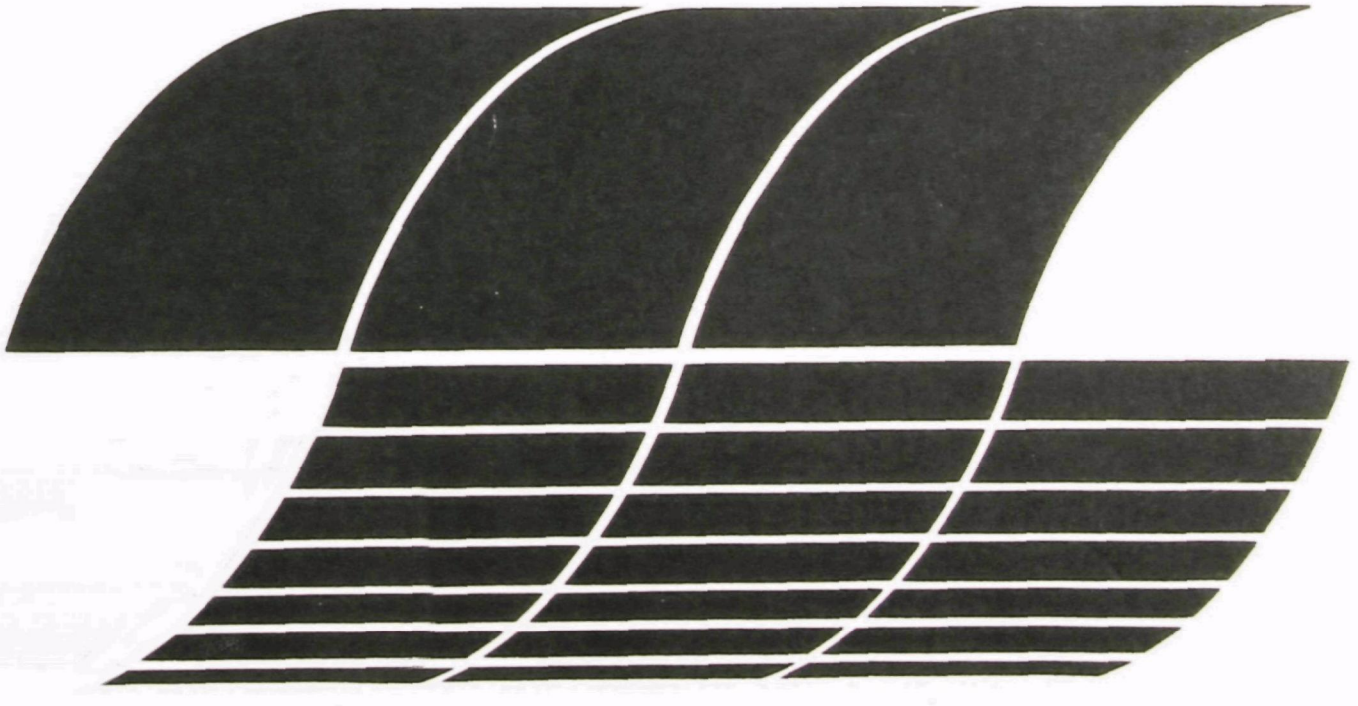
Guidelines for Particulate Sampling in Gaseous Effluents from Industrial Processes

Interagency Energy/Environment R&D Program Report



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EPA REVIEW NOTICE

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Guidelines for Particulate Sampling in Gaseous Effluents from Industrial Processes

by

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ABSTRACT

This guideline document lists and describes briefly many of the instruments and techniques that are available for measuring the concentration or size distribution of particles suspended in process streams. The standard, or well established, methods are described as well as some experimental methods and prototype instruments.

Descriptions of instruments and procedures for measuring mass concentration, opacity, and particle size distribution are given. Procedures for planning and implementing tests for control device evaluation are also included.

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ACKNOWLEDGMENT

Members of the Southern Research Institute staff who reviewed this report are Joseph D. McCain, Charles Feazel, and James Ragland. We appreciate the suggestions and continuing support of D. Bruce Harris, our Project Officer.

SECTION I

INTRODUCTION AND SUMMARY

The purpose of this guideline document is to describe the instruments and techniques that are available for measuring the concentration or size distribution of particles suspended in gas process streams. The standard or well established methods are described as well as some experimental methods and prototype instruments. A more detailed discussion of particulate sampling methods is given in a companion document "Technical Manual: A Survey of Equipment and Methods for Particulate Sampling in Industrial Process Streams", EPA report number EPA-600/7-78-043, March, 1978, by Wallace B. Smith, Paul R. Cavanaugh, and Rufus R. Wilson.

It is disappointing to everyone involved in aerosol sampling that more convenient and efficient methods are not available for making measurements of particle size and concentration. When good resolution and accuracy are needed, one must rely on manual techniques such as filters for mass and cascade impactors for sizing measurements. Nevertheless, progress is being made in the development of more convenient methods that yield real-time information. For some applications, such instruments already yield useful information. Table I summarizes the current status of particulate sampling methods.

Section II contains descriptions of instruments and procedures for measuring mass concentrations, Section III is devoted to measurements of opacity, Section IV to particle-size measurements, and Section V specifically to control device evaluation.

TABLE I.

STATUS OF PARTICULATE SAMPLING METHODS FOR PROCESS STREAMS

MASS CONCENTRATION	
Filters - C	Transmissometers - P
β-Particle Attenuation - P	Light Scattering - P, CP
Charge Transfer - CP	Piezoelectric Microbalances - R
OPACITY	
Transmissometers - C Nephelometers - CP	
PARTICLE SIZE	
Cascade Impactors - C, P	Diffusion Batteries and Condensation Nuclei Counters - P
Cyclones - P, C	
Light Scattering - P	Electrical Mobility - P

C - Commercial instruments in everyday use.
CP - Commercial instruments available, these may require special adaptation or skills.
P - Prototype systems have been used. These require special adaptation or skills.
R - Established measurement techniques, but not applied to process streams.

SECTION II

PARTICULATE MASS CONCENTRATION

FILTRATION

Particulate mass concentration measurement methods using filtration as the means of sample collection can be classified according to the sampling flow rate used and the location of the filter in or out of the gas process stream. Low sampling flow rate methods usually sample in the 14.2 ℓ/min (1/2 ft^3/min) to 42.5 ℓ/min (1 1/2 ft^3/min) range. High flow rate methods usually operate above 142 ℓ/min (5 ft^3/min). Use of a filter located outside the process stream is referred to as an extractive method. Use of the filter located in the process stream is referred to as an in situ method.

Various organizations have promulgated specific procedures and sampling train designs for one or more of these methods. The EPA Test Method 5[1] specifies the use of extractive sampling and EPA Test Method 17 specifies the use of in situ sampling.[2] The American Society for Testing Materials (ASTM) specifies an in situ sampler.[3] The American Society of Mechanical Engineers (ASME) Performance Test Code 27 specifies the use of either an in situ or extractive sampler.[4] The ASME will soon be releasing a new Performance Test Code 38 which will supercede the Performance Test Code 27.

EPA Test Method 5

Compliance testing of stationary sources for particulate emissions must be conducted with the EPA Test Method 5, "Determination of Particulate Emission from Stationary Sources".[1] The stationary sources covered include new steam boilers, incinerators, cement plants, and pulp and paper mills. All states require the use of some form of the Method 5 train for compliance testing. Method 5 relies on the removal or extraction of a dust laden gaseous sample from the duct or stack followed by the subsequent removal of the particles onto a filter media with concurrent measurement of the sample volume to determine particulate mass concentration. Since the filter must be kept at 120 ± 14°C, the particulate mass includes any material that condenses at or above the filtration temperature. The particulate concentration is found for the dry component of the stack gas, omitting the amount contributed by water and other vapors. Finally, this is expressed as the concentration that would be present under conditions of standard temperature and pressure.

A sample is removed from the duct by using a prescribed traversing procedure which involves drawing portions of the sample from different points within the duct. Isokinetic sampling conditions must be maintained; that is, at each traverse point, the sample velocity at the nozzle is adjusted to equal the duct velocity. This procedure yields, in effect, an approximate integration of collected mass and sample volume over the cross-sectional area of the duct.

The Federal Register[1] gives detailed specifications for the apparatus comprising the sampling train which must be used to properly conduct a Method 5 test. The sampling train consists of a nozzle, probe, pitot tube, particulate sample collector,

gaseous sample collector, sampling box, and meter set (refer to Figure 1). The user can either construct his own sampling train by following the specifications[5] or he can use one of the many commercial models available (see Table II). A recent collaborative test showed a trained crew could be expected to produce results with a standard deviation of 12%.[6]

An inherent limitation of the Method 5, indeed, of all stack sampling systems, is the inability to obtain particulate matter in the same state as it exists when the plume mixes with the atmosphere. This change to atmospheric conditions may result in particulate matter being formed in the plume that was not present in the stack.

EPA Test Method 17

EPA Method 17 uses an unheated in-stack filter and probe with the basic sampling train design of Method 5 to sample particulate emissions isokinetically. It is appropriate for situations where particulate mass concentrations are independent of temperature and where the gaseous effluents are not saturated with water. Determination of compliance with new source performance standards can be made with Method 17 when it is specifically provided for in a subpart of the standards.[2]

ASTM - Test Method

Both the ASTM and the ASME provide specifications for in situ samplers. The ASTM Method is similar to the EPA Test Method 5, the main difference is the use of an instack filter. However, the sizes of the sampler components (tubing, filter holder, etc.) usually place an upper limit on the flow rate. With the ASTM arrangement, shown in Figure 2, a thimble-shaped filter is used to sample high mass concentrations. The pitot tube, pump, and other parts are similar to the Method 5 sampler.

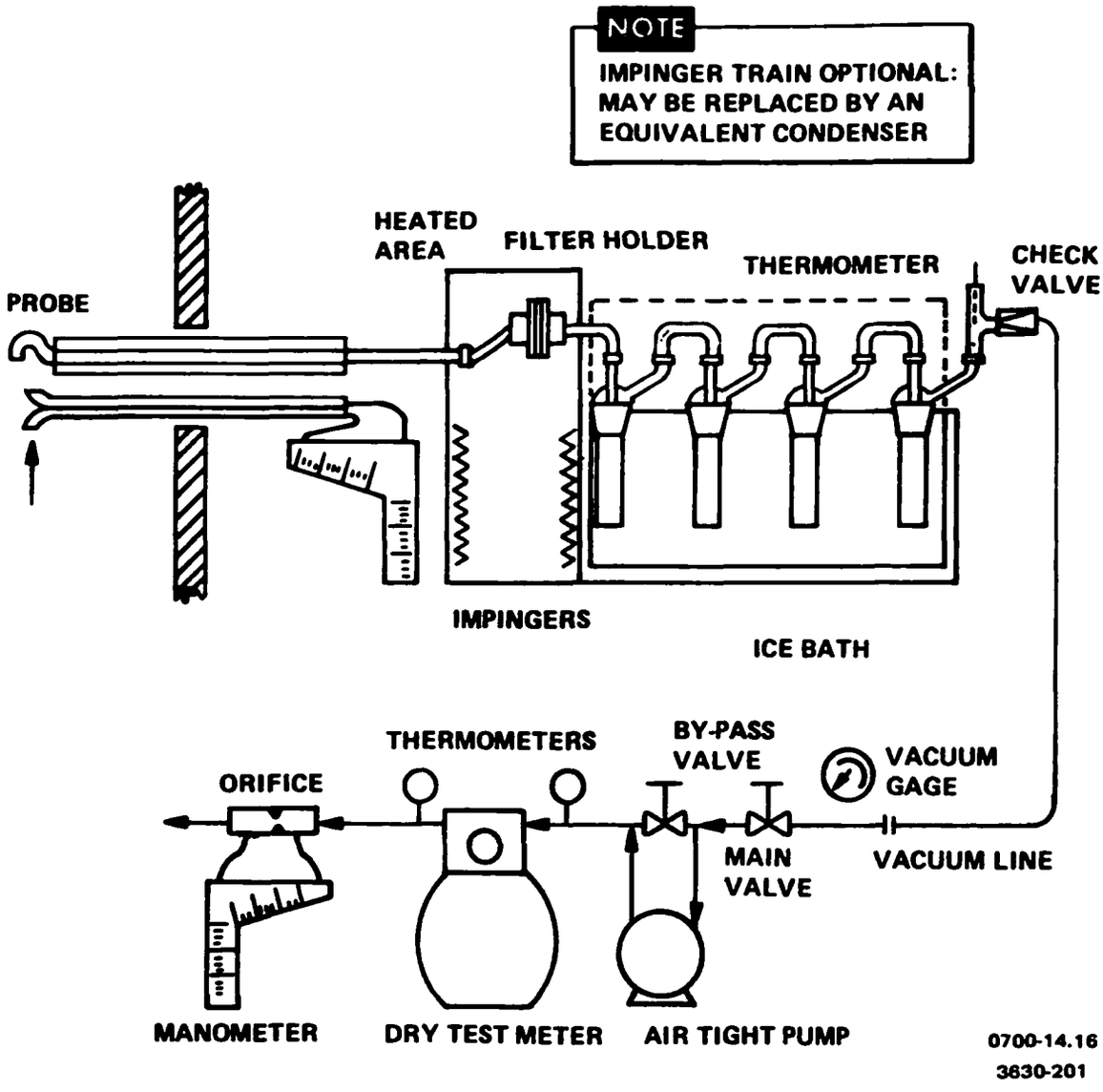


Figure 1. The EPA Method 5 particulate sampling train.

TABLE II. SAMPLING SYSTEMS FOR TESTING BY EPA METHOD 5

Company	Address	Train Title
Aerotherm-Acurex	485 Clyde Avenue, Mountain View, CA 94042	High Volume Stack Sampler
Glass Innovations, Inc.	P.O. Box B Addison, NY 14801	The Source Sampler
Joy Manufacturing Co.	Commerce Road Montgomeryville, PA 18936	Emission Parameter Analyzer
Lear Siegler, Inc./ Environmental Technology Div.	One Inverness Dr. East Englewood, CA 80110	PM100 Manual Stack Sampler
Misco International Chemicals, Inc.	1021 S. Noel Avenue Wheeling, IL 60090	Stack Source Sampler
Research Appliance Company	Pioneer and Hardies Rd. Gibsonia, PA 15044	Staksamplr
Scientific Glass & Instruments, Inc.	7246 Wynnewood Houston, TX 77001	Stack-O-Lator

Note: Most companies will supply filters for use with their trains upon request.

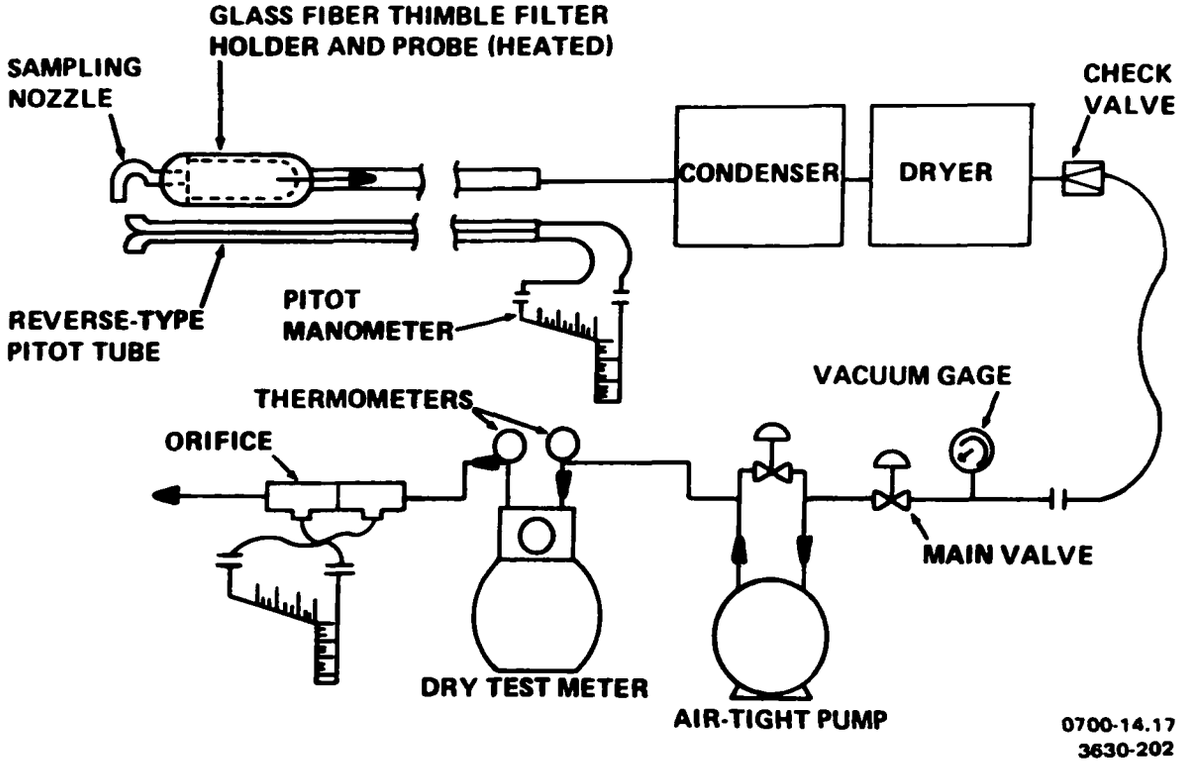


Figure 2. ASTM type particulate sampling train.

ASME Performance Test Code 27

The ASME Performance Test Code allows the use of a variety of instruments and methods.[4] Paragraph 55 of Section 4 of the Code states "Testing experience has not been uniform enough to permit standardized sampler design. This code, therefore, merely gives limiting requirements which past experience has shown desirable to avoid major sources of error". The Code is designed as a source document which provides technically sound options to be selected and agreed upon by the sponsor and the contractor who performs the sampling.

Advantages and Disadvantages

The main advantage of the in situ mass sampler is that substantially all of the particulate matter is deposited directly in the filter and some in the nozzle; therefore, only the nozzle and filter holder need to be washed. Because the filter is maintained at the stack gas temperature, auxiliary heating of the filter is usually not needed.

The main disadvantage is that the in situ sampler is limited to process streams with temperatures that do not exceed the limit of the filter media and holder, and low moisture levels. Thermal expansion of the filter holder may create gas leakage. Also, the instack filter cannot yield data on the particulate fraction due to cooling, e.g. in the plume.

Filter Materials

Filter materials for use in particulate collection equipment are available from scientific equipment supply houses in several different shapes, sizes, and compositions. Although membrane, cellulose, metal-alloy, quartz, and ceramic filters are available, the most widely used for stack sampling is the glass fiber filter. Glass fiber filters commonly used in air pollution mea-

TABLE III. GLASS FIBER FILTER PRODUCTS

Company	Address	Filter Name
Balston, Inc.	703 Massachusetts Avenue Lexington, MA 02173	Balston Microfibre
Gelman Instrument Company	600 S. Wagner Road Ann Arbor, MI 48106	Gelman Type A Gelman Type AE Spectrograde
Mine Safety Appliance Company	400 Penn Center Blvd. Pittsburgh, PA 15235	MSA 1106-BH
Whatman, Inc.	9 Bridewell Place Clifton, NJ 07014	GF/A, GF/C, GF/D Reeve Angel 900AF Reeve Angel 934AH

surements are listed in Table III. This list is not exhaustive. For a particular test, a filter should be chosen considering the objectives of the testing program and the characteristics of the sampling environment and equipment.

PROCESS MONITORS

The ideal process stream mass monitor would have the following features:

1. The sensing principle used to detect the particles in a gas stream would be a direct measurement of the mass of the particles.

2. The mass sensor would be insensitive to such factors as changes in gas temperature and humidity, corrosive gases, and liquid droplets.

3. The monitor would provide continuous, instantaneous ("real-time") measurements of mass concentration.

4. Since the mass concentration in a process stream often varies over the cross-sectional area of the duct, the ideal monitor would measure the average mass concentration across the entire cross-sectional area of the duct.

5. A monitor with its sensor mounted directly within the gas stream, called an in situ monitor, is generally preferred over the extractive monitor, in which the sample may be altered significantly prior to the measurement.

No monitor currently available has all the above qualifications. The development of process monitors has begun to gather

momentum only recently, and much of the performance data pertaining to their operation at various sources and under various conditions has been shown to be contradictory or of limited usefulness. Nevertheless, a process monitor may provide sufficient accuracy for certain applications.

Beta Radiation Attenuation Monitors

When beta particles impinge on matter, some are absorbed, some are scattered, and some are transmitted. The reduction in the incident beam intensity in passing through the sample is known as beta radiation attenuation. Beta radiation attenuation is practically independent of the chemical composition of the absorber and thus is considered by many engineers and scientists to be a direct measure of mass.

Current instruments use either a Carbon-14 or a Promethium-147 source; a Geiger-Müller, proportional counter, scintillation, or solid state detector; and a filter, cyclone, combination cyclone-filter, or an electrostatic precipitator collector. Recent models include computerized data reduction and digital display of mass concentration. The temperature limit is 538°C (1000°F) with the use of a sample diluter and conditioner such as the prototype shown in Figure 3. Although beta monitors of several designs have been tested on industrial sources over the past ten years, very little information is available on their performance, and they must still be considered prototype devices.

Advantages include a sensing principle that is very closely correlated to mass and independent of particle composition, low sensitivity to particle and aerosol parameters other than mass, and a movable filter tape which makes it convenient for performing chemical analysis concurrent with sampling.

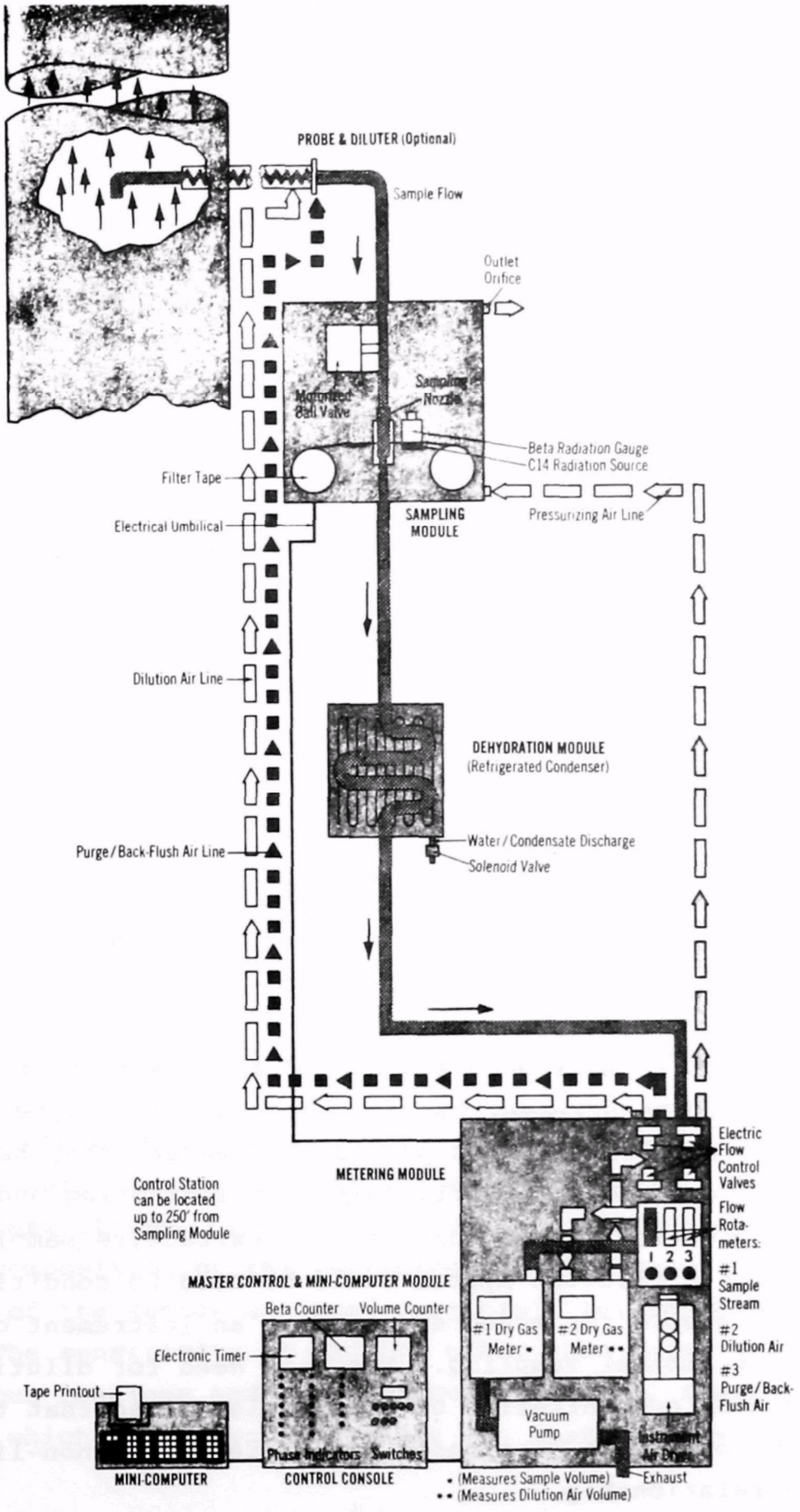


Figure 3. Schematic flow diagram of a prototype Automatic Stack Monitor System installation. (Drawing not to scale.) Used by permission of Research Appliance Company.

Disadvantages include a response time longer than some other monitors, the need for an extraction/dilution system, and a sensitivity to variations in filter tape thickness. Errors may result from sample losses in the probe, variations in the filter tape thickness, nonuniform deposition of dust on the tape, variations in particle collection efficiency, statistical variations in the count rate, and variations in the predicted count rate due to the nonexponential character of beta radiation attenuation.

Piezoelectric Mass Monitors

Piezolectricity is a property of certain crystals, such as quartz, which involves the production of an electrical charge on certain faces of the crystal when the crystal becomes mechanically stressed. The converse process also occurs; that is, a piezoelectric crystal becomes mechanically stressed where an electrical charge is placed on certain faces. This two-way capability is responsible for the ability of a piezoelectric crystal to cause an oscillating electric circuit to oscillate at the natural vibrational frequency of the crystal. When foreign material, such as aerosol particles, adheres to the surface of a vibrating piezoelectric crystal, the natural frequency of vibration of the crystal decreases. The magnitude of the frequency change is directly proportional to the mass of the added material.

Piezoelectric monitors have had no applications in sampling industrial process streams, nor are there any prototype monitors known to be designed for this purpose. However, they have been used for ambient and automobile emissions monitoring and show promise as process stream monitors. An extractive sampling system combined with a dilution system could be used to condition a process stream sample for measurements with an instrument originally designed for ambient sampling. The main need for dilution is to lower the mass concentration of the sample gas so that the sensor of the monitor is not overloaded, thus yielding a non-linear mass-frequency relationship.

Advantages include a sensing principle that relates directly to mass and which is independent of particle composition, and yields continuous, instantaneous ("real-time") measurements.

Disadvantages include a need for an extraction/dilution system, sensitivity to changes in gas temperature and humidity (decreases for particle sizes greater than 2 μm), and the necessity of periodic cleaning to prevent non-linear sensor response.

Charge Transfer

The transfer of an electric charge occurs when two bodies of different composition come into contact. The transfer can occur during either static contact or triboelectric (rubbing) contact. The mechanism of transfer in static contact is essentially the same for metals and semiconductors.[7,8]

In all charge-transfer instruments, the aerosol stream is forced to collide with a sensor. When the particles in the aerosol stream contact the sensor, a charge is transferred producing a current that is continuously monitored with an electrometer. Since the amount of charge transferred is proportional to the mass of the particle which collided with the sensor, the electrometer can be scaled to read the mass concentration.

There are other factors, beside mass, that can affect the amount of charge transferred to the sensor from particles in any given process stream. Some of the possible factors are: the chemical composition of the particles and the sensor material, condition of the sensor surface, particle size, and particle charge. The sensitivity to factors other than mass can result in erroneous readings and frequent need for recalibration. The extent to which these factors affect the instrument's response

is discussed in a paper on a laboratory study of the IKOR Model 206.[9] The IKOR Air Quality Monitors (IKOR Inc.), P.O. Box 660, Blackburn Industrial Park, Gloucester, Massachusetts 01930, use a bullet shaped Inconel metal sensor. They are available in three models. Models 206 and 207 are extractive; Model 2710 is the newly developed in situ monitor.

Charge transfer monitors have been used on industrial sources for over 14 years. Advantages include in situ or extractive sampling and continuous, instantaneous, real-time measurements. Disadvantages include indirect measurement of mass; strong dependence on chemical composition of the particles; sensor sensitivity to particle size (suspected lower size limit due to low impaction probability for small particles), water droplets, corrosive gases, and particle charge; and degradation of sensor performance when exposed to wet, waxy or sticky particles which coat the sensor. This last disadvantage would hamper usage at combustion systems fired with residual oil. Sources with electrostatic precipitation present precharging problems, as discussed. In conclusion, the IKOR monitor performs best when applied to the situation where process stream conditions are constant or change predictably, and which contain dry, discrete, uncharged particles.

Optical Methods

Conventional Transmissometers--

Light scattering theory predicts a dependence of light attenuation not only on mass concentration but also on particle size and composition. Figure 4 shows the results of applying this theory to calculate the effects of various particle sizes

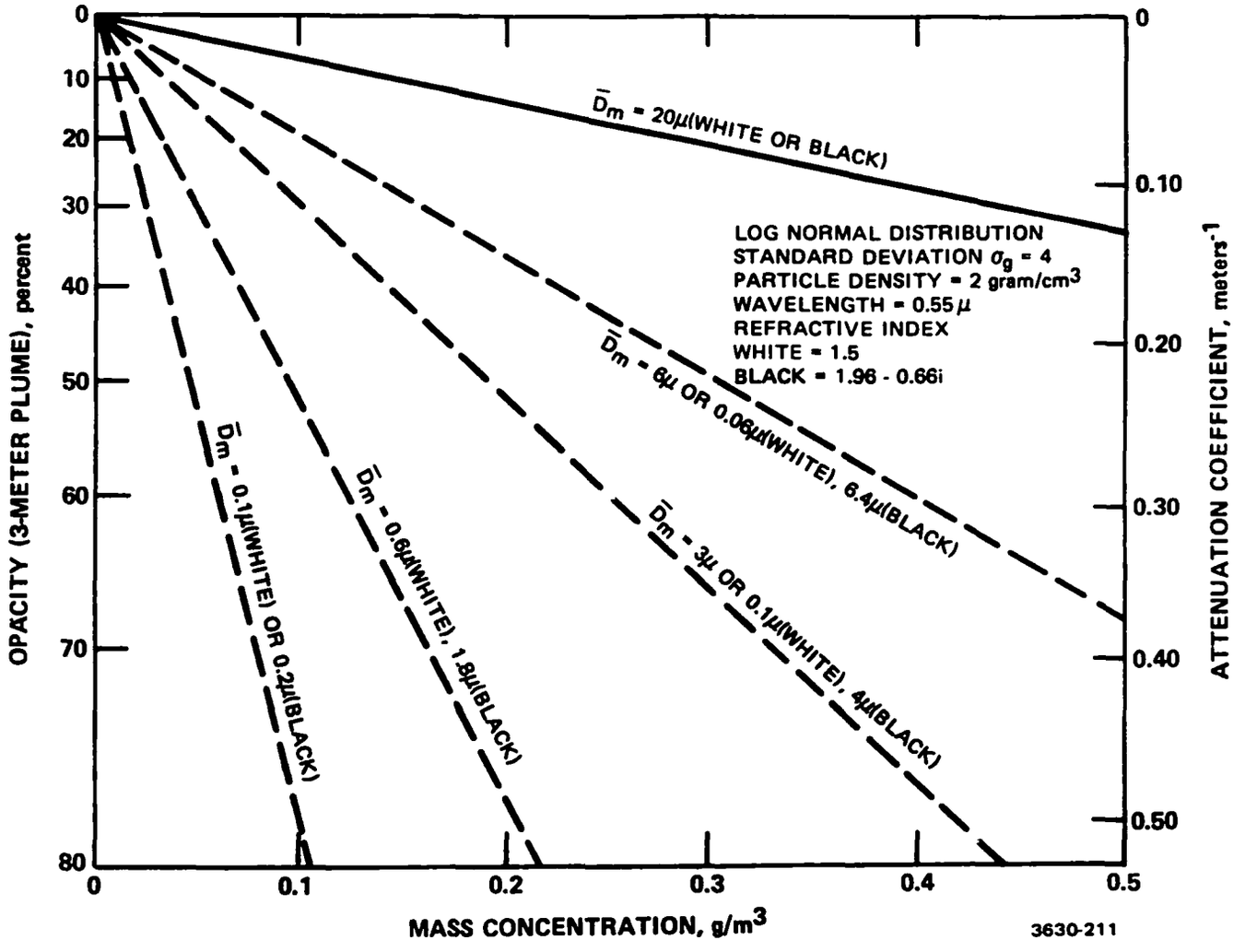


Figure 4. Opacity of smoke plumes containing particles of different sizes and refractive indexes as a function of their mass concentration. After Connor.[10]

and composition on the relationship between the opacity and mass concentration of aerosols.[10] At particle diameters above 3 or 4 μm the refractive index of the particle plays little role in determining the opacity-mass concentration relationship. However, at particle diameters below 3 or 4 μm, the refractive index plays a major role.

For a transmissometer to be useful as a monitor of the mass concentration, the properties (other than mass) of the particles being monitored must remain fairly constant over the monitoring period. Experimental data are available showing that good opacity-mass concentration calibration can be obtained on some sources. The sources evaluated include coal-fired plants;[11,12,13] lignite-fired power plants;[14] cement plant;[15] Kraft pulp mill recovery furnace;[16] petroleum refinery, asphaltic concrete plant, and a sewage sludge incinerator.[17] Once calibrated, transmissometers may be useful indicators of mass emissions on sources where the aerosol properties remain constant.

Conventional transmissometers are routinely used for providing a qualitative measurement, i.e., where changes in opacity are used as a general indicator of changes in mass concentration. Generally, transmissometers are not relied upon to produce quantitative measurements; i.e., where actual values of mass concentration are obtained. This is due to the uncertainty introduced by the strong dependence of the sensing principle on the particle size distribution and index of refraction. The transmissometer does possess the advantage of being able to provide an in situ, continuous, real-time, integrated measurement. In conclusion, it is unlikely that conventional transmissometers will ever be used for routine quantitative measurement of mass concentration. The multiple-wavelength transmissometer, discussed in the next subsection, is a better candidate because it eliminates the uncertainties caused by variable particle size distribution.

Other Optical Methods--

Multiple-wavelength transmissometers--The general principle underlying the multi-wavelength transmissometer can be seen by referring to Figure 5. In this figure, the mean extinction coefficient ($\bar{E}$) is shown as a function of the phase shift parameter (ρ_{vs}) of a polydisperse aerosol.[18]

By making measurements of optical transmittance (opacity) at two or more well separated wavelengths, points on a response curve like the one shown in Figure 5 are obtained. Two such points are sufficient to determine the average particle size and the particulate concentration in an aerosol.[19] The confusion introduced into measurements of transmittance by variations in particle size is removed by this technique, but the dependence on refractive index remains.

To measure the transmittance as a function of wavelength, the wavelength must be varied. This can be accomplished with white light and monochromatic filters or a monochromator, or with multiple laser sources. The system records the transmission through the stack at each of the selected wavelengths.

There are several complex computational methods whereby the particle size distribution and mass concentration can be obtained from the optical density measurements made at the different wavelengths. These are discussed in detail by Kerker.[20]

The use of multiple-wavelength transmissometers to monitor mass emissions seems promising, but the systems are more complicated than ordinary transmissometers, and an undesirable dependence on the particle refractive index can introduce errors.

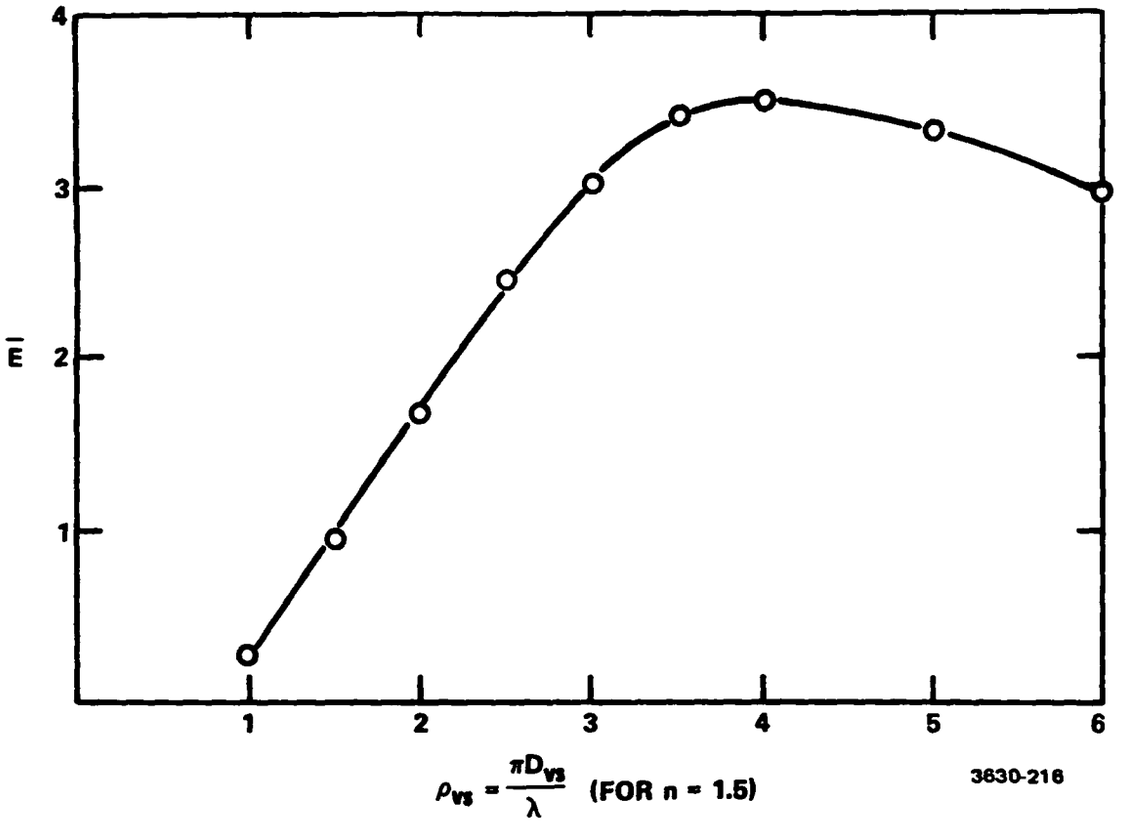


Figure 5. Mean extinction coefficient as a function of the phase shift parameter ρ_{VS}. After Dobbins and Jizmagian.18

Light scattering--Suspended particles in an aerosol will scatter (diffract, refract, and reflect), and absorb incident light; the remaining portion is transmitted. Whereas transmissometers use this remaining portion of the incident light as a measure of the particulate mass concentration or of opacity, other instruments use the scattered portions. Instruments that detect the scattered light can be much more sensitive at low particulate concentrations than transmissometers.

Nephelometers, devices that attempt to measure all of the scattered light, have recently been applied to stack monitoring. One such instrument, the Plant Process Visiometer (PPV), has been developed by Meteorology Research, Inc.[21,22,23] This instrument is designed to measure opacity and is not considered a mass monitor per se; however, it is normally quite sensitive to mass changes. A diagram of its optical assembly is shown in Figure 6. The sample, extracted through a probe with no dilution, is passed through the detector view.

An in situ monitor has been developed[24] that is based on the measurement of the backscattered light. It uses a laser as the light source and is a single ended instrument, i.e., both the light source and detector are located within the same enclosure. The instrument is the PILLS V (see Figure 7). It and the improved model P-5A is a member of a family of Particulate Instrumentation by Laser Light Scattering devices developed by Environmental Systems Corporation. At present, the instrument does not possess the capability to traverse large stacks in order to obtain multi-point measurements. Since the particulate mass concentration is frequently not uniform across the entire cross-sectional area of the stack, the use of such a small sampling volume and the inability to traverse creates a problem when trying to obtain data that is representative of the actual total mass concentration present within the stack.

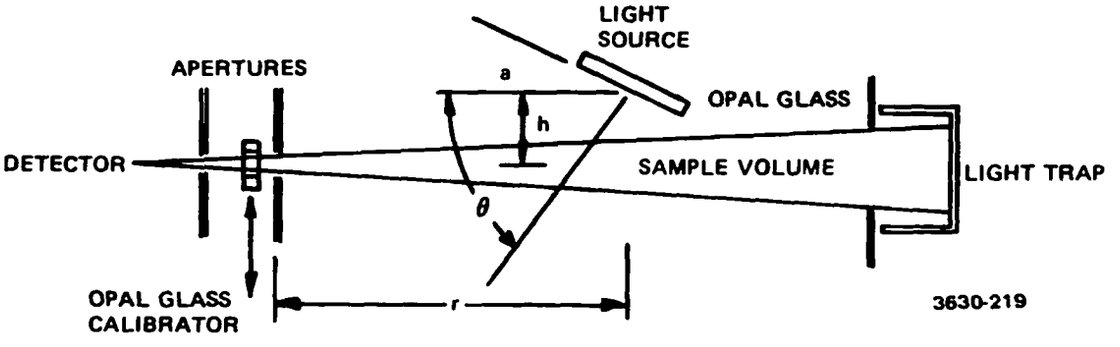


Figure 6. Optical assembly diagram of a nephelometer used in stack monitoring. After Ensor and Bevan.[21]

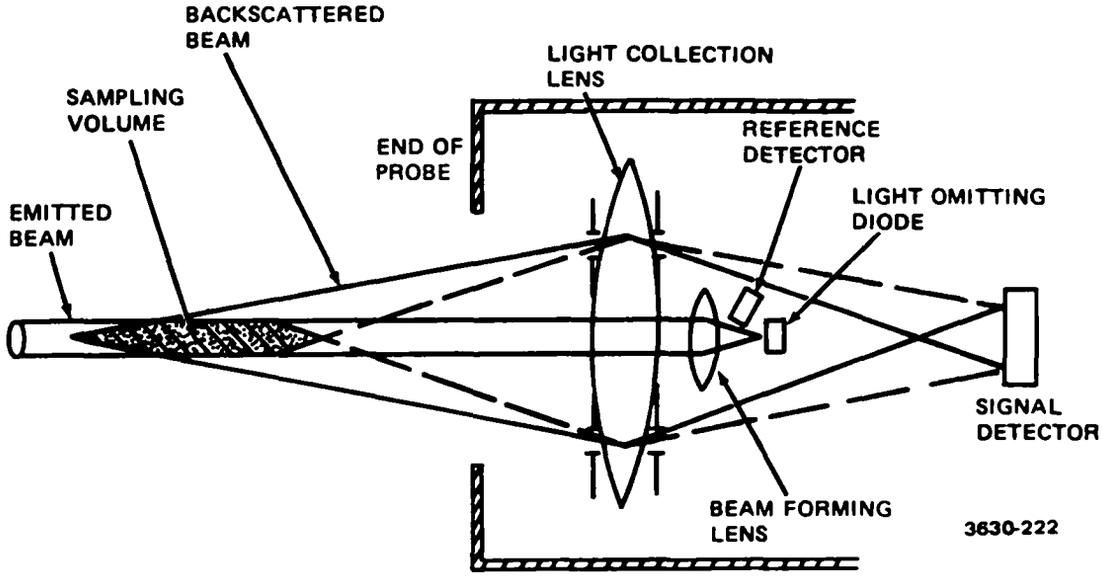


Figure 7. Optical diagram of the PILLS V instrument. After Schmitt, Nusplinger, and Kreikelbaum.[25]

Light scattering instruments suffer from some of the same problems as transmissometers when attempting to infer mass; i.e., sensitivity to particle size, shape, and chemical composition. The functional dependence of the instrument response to these factors is determined by the detection angles employed relative to the incident beam. The effects of such behavior are accounted for in practice by calibrations of the instrument against another more direct mass measurement of the aerosol of interest.

SECTION III

OPACITY

Suspended particles in an aerosol will scatter and absorb radiation; the remaining portion is transmitted. The transmittance, T, of a fluid medium containing suspended particles is defined as the ratio of transmitted radiation intensity to incident radiation intensity. T is given by the Bouguer, or the Beer-Lambert, law:[27]

$$T = \exp(-EL) \tag{1}$$

where L is the thickness of the medium, and E, the extinction coefficient of the medium, is a complicated function of the size, shape, total projected area, refractive index of the particles, and the wavelength of the radiation.

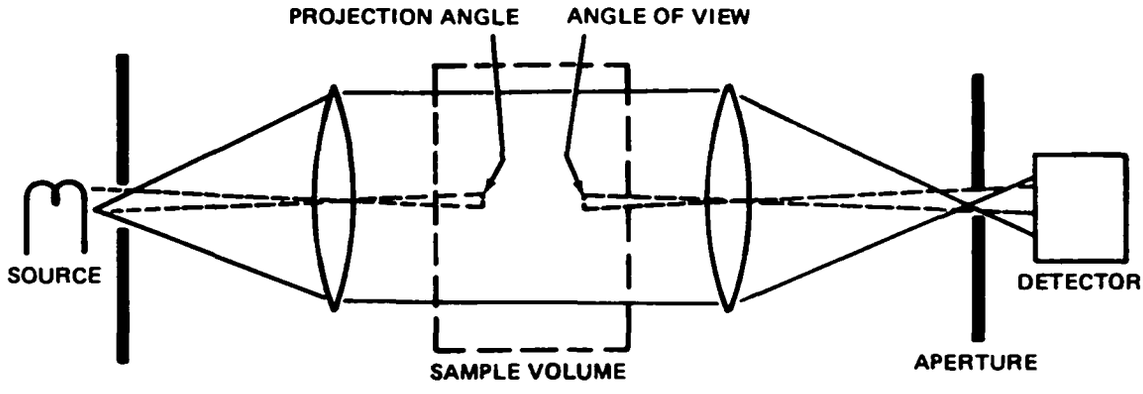
While transmittance is defined as the ratio of the intensity of the light transmitted through the aerosol to the intensity of the incident light, opacity is defined as the ratio of the intensity of the light attenuated by the aerosol to the intensity of the incident light (i.e., opacity = 1/T). Aerosols which transmit all incident light are invisible, have a transmittance of 100%, and an opacity of zero. Emissions which attenuate all incident light are totally opaque, have an opacity of 100% and a transmittance of zero. By definition, opacity can only be measured rigorously using transmittance, rather than light scattering measurements, because the latter yield no measure of the quantity of light that is absorbed.

As the opacity, 1-T, approaches zero, the relative error in its value as calculated from a measurement of transmittance becomes unavoidably large. For example, a two percent error in a transmittance measurement of 98% gives a 100 percent error in the calculated value of opacity. In such cases a nephelometer as used by Ensor,[22] may be a more accurate measure of opacity although it requires a probe and sampling traverses.

To obtain true transmittance data the collimation angles (angles of view and projection) for the transmitter and receiver must be limited to reduce the sensitivity to stray light scatter (see Figure 8). A zero degree angle is the ideal collimating angle, whereas a finite angle will introduce a systematic error. However, a compromise is necessary, since as a zero degree collimation is approached, instrument construction costs, operating stability, and optical alignment problems increase.

Many versions of transmissometers, or smoke meters, are available as stack emission monitors. If the transmissometer is used to measure in-stack opacity for purposes of compliance with federal regulations, it must meet the EPA requirements for opacity measurement systems as specified in the Federal Register of September 11, 1974.[28] For instance, the use of visible light as a light source is required. For other uses of the data, it may be possible to operate with nonvisible wavelengths. The angle of view and the angle of projection are both specified, for compliance, as no greater than 5° (see Figure 8).

A typical double pass in situ transmissometer design is illustrated in Figure 9. The design shown employs a chopped, dual-beam, optical system that automatically compensates for the effects of temperature, voltage changes, and component aging.[30] The same source is optically divided into a measuring beam and a reference beam. The measuring beam is reflected back by a



SCHEMATIC OF A TYPICAL TRANSMISSOMETER SYSTEM

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Figure 8. Schematic of a transmissometer showing projection and view angles which must be no greater than 5° for EPA compliance.

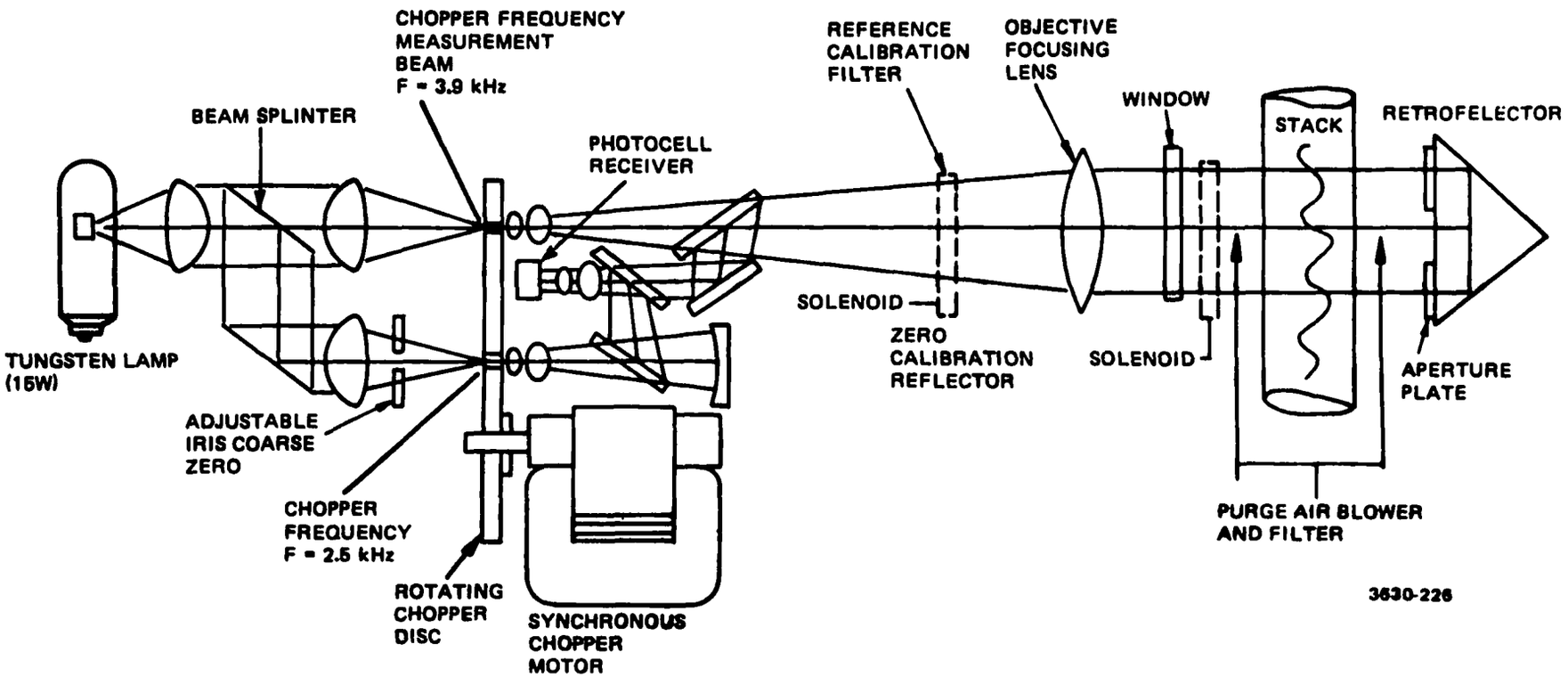


Figure 9. A typical double pass in situ transmissometer design. After Nader.[29]

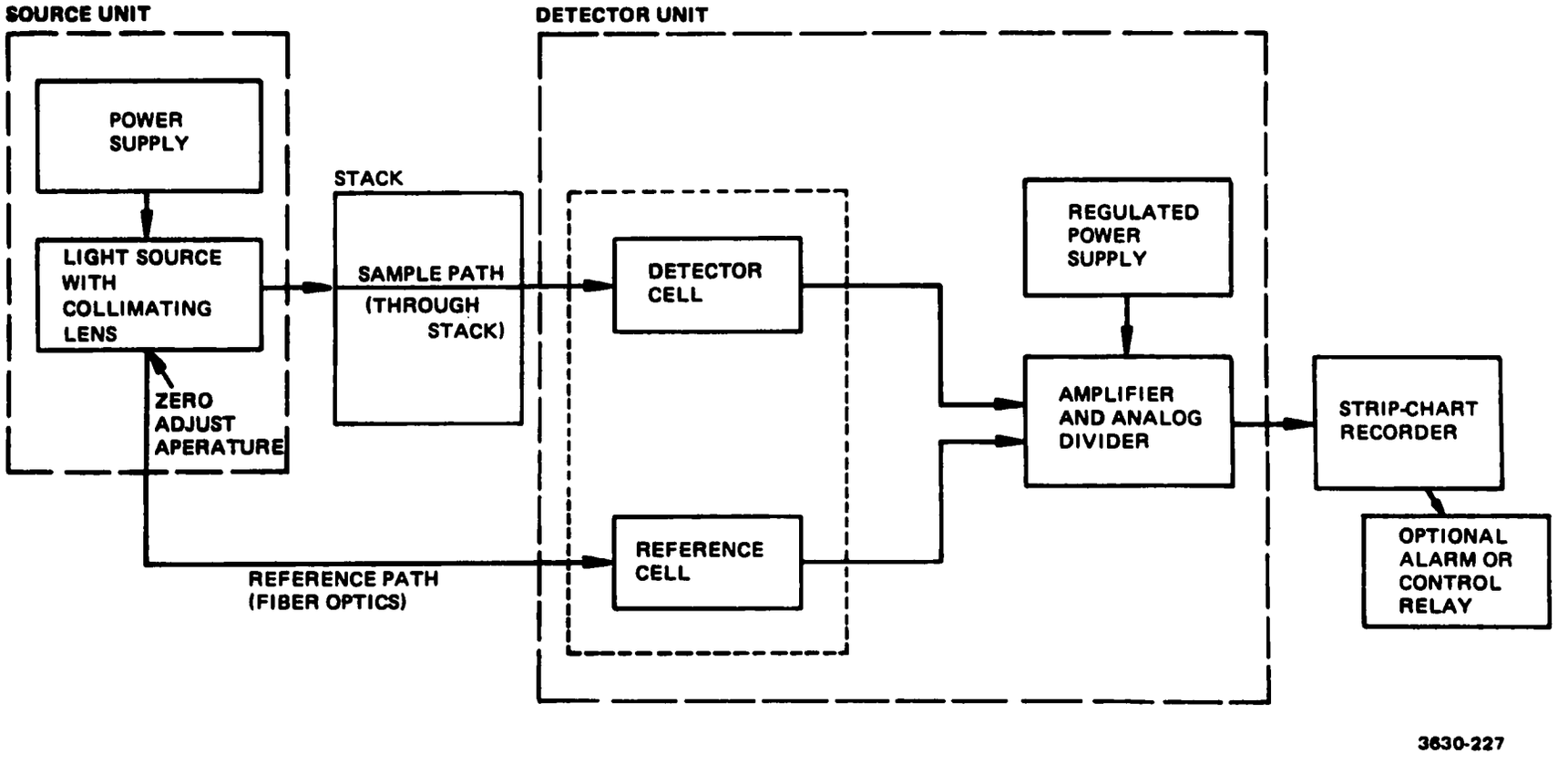
corner-cube retroreflector. The reference beam provides automatic gain control to compensate for any changes in detector response or source intensity. Both the transceiver and retroreflector unit are specially constructed with air purging attachments to keep the optical windows free of particulate deposits, and can provide adequately clean windows for three or more months of unattended operation.

A block diagram of a single pass transmissometer design is shown in Figure 10. In this design, the light source with the collimating lens and power supply are placed on one side of the stack with the detector cell, electronics and power supply on the opposite side. The beam makes only one pass through the stack gas which eliminates the problems caused by reflectivity or back scattering of the effluent being measured.

Transmissometers usually contain an alarm or warning system that alerts plant personnel when the opacity exceeds a preset limit. An alarm and/or plant cut off switch can be automatically activated when limit values are exceeded. One instrument possesses the ability to integrate the opacity measurements over various time intervals. This permits automatic monitoring and control of unacceptable emission or dust levels which are present for long periods of time, and not for just a brief moment.

Comparisons of transmissometer measurement with visual plume opacity have been made, and have generally shown favorable results. The in-stack measurement is usually compared with an out-of-stack plume measurement performed by visual observation by a trained observer or performed by telephotometry.

Besides the collimating angles of the transmissometer, the important parameters affecting transmissometer performance in a given process stream are the particle size distribution in the process stream, particle shape and refractive index, and the wave-



FUNCTIONAL BLOCK DIAGRAM

Figure 10. A single pass transmissometer design. After Haville.[31]

length of the transmitted radiation. The effect of these parameters is reflected in the measured values of the mean extinction coefficient, E, of the process stream. The mean extinction coefficient, E, can be determined by summing the particle extinction coefficients, Q_E, of the particles in the process stream. Figure 11 gives the theoretical extinction coefficient for spherical particles with typical indices of refraction (relative to air) of 1.3 to 1.6 under white light illumination. For particles in the Rayleigh scattering region (diameter (d)<0.05 μm), Q_E is approximately zero. For those in the Mie scattering region (0.05 <d<2 μm)) Q_E varies from 0 to 4. For those particles in the geometric scattering region (d>2 μm) Q_E approaches a theoretical limit of 2 for very large particles.

In practice, the particles in stack emissions are polydisperse and the incident light is polychromatic. This results in a smoothing out of the oscillatory behavior depicted in Figure 11. However, a size distribution of transparent particles which mostly lies within a narrow range of particle sizes in the Mie region can result in transmittance measurements yielding opacity values similar to opacity values for much higher mass concentrations of absorbing particles.

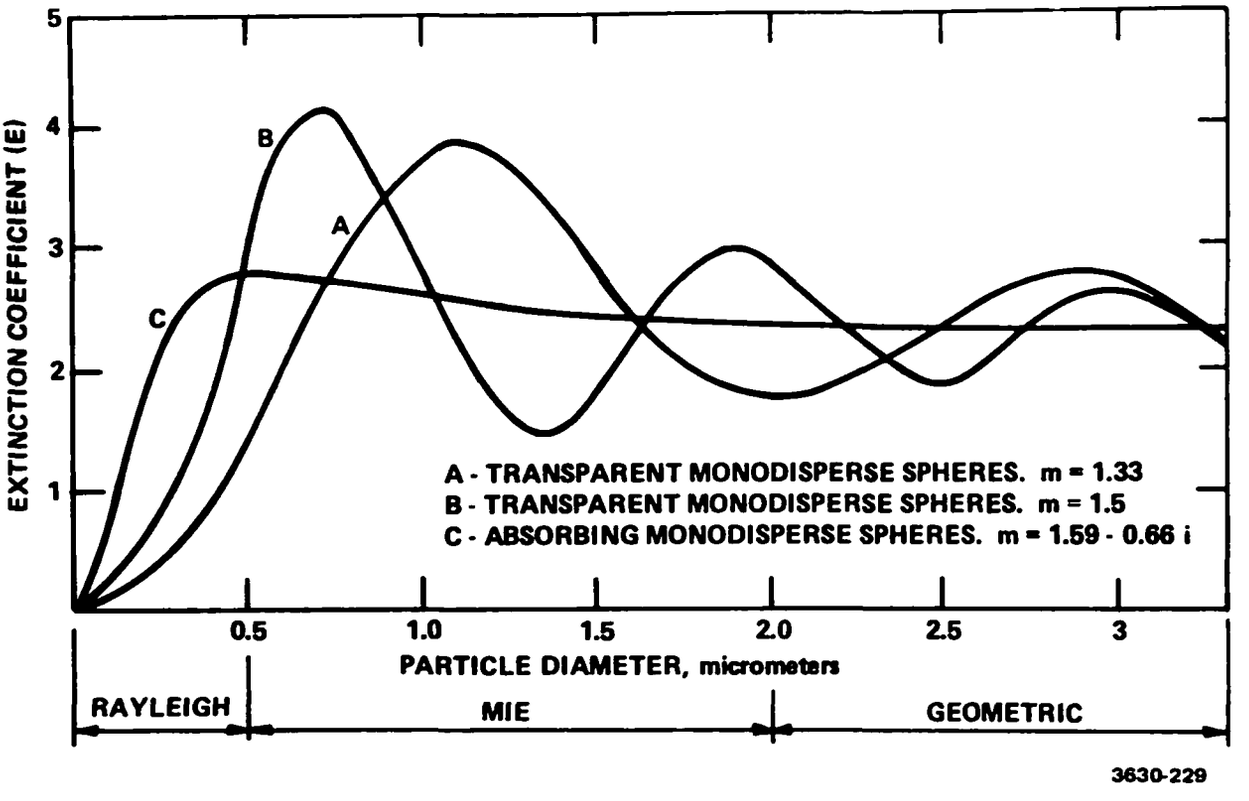


Figure 11. Particle extinction coefficients for various aerosols over three scattering regions: Rayleigh, Mie, and Geometric. After Hodkinson and Greenfield.[32]

SECTION IV

PARTICLE SIZE DISTRIBUTIONS

The methods of sizing particles can be classified as either established, widely used techniques or new techniques which have not yet received widespread use.

ESTABLISHED TECHNIQUES

The established techniques of particulate sizing can be divided into those that size particles at the test site and those that require a sample to be collected at the test site and examined in a laboratory environment. Often the laboratory measurements require the dust to be redispersed.

Field Measurements

On-site particle sizing instruments classify particles by using aerodynamic (inertial), optical, diffusive, or electrical mobility methods.

Aerodynamic Methods--

In order to avoid unnecessary complications in data presentation, particles of different shapes may be assigned aerodynamic diameters. The aerodynamic diameter of a particle is the diameter of a unity density sphere that has the same settling velocity as the particle of interest. The aerodynamic diameter is related to the way that a particle will behave in the respiratory system as well as in aerodynamic sizing devices.

Examples of aerodynamic particle sizing instruments are centrifuges, cyclones, cascade impactors, and elutriators. Each

of these instruments employs the unique relationship between a particle's diameter and mobility in gas or air to collect and classify the particles by size. For pollution studies cyclones and impactors, primarily the latter, are more useful because they are rugged and compact enough for in situ sampling. As previously explained, in situ sampling is preferred because the measured size distribution may be seriously distorted if a probe is used for sample extraction. In the following two subsections, methods of using impactors and cyclones are discussed.

Cascade impactors--Because of its compact arrangement and mechanical simplicity, the cascade impactor has gained wide acceptance as a practical means of making particle size measurements in flue gases. Their long-time use as ambient samplers has resulted in a large number of experimental studies on cascade impactor design and performance in the laboratory.[33,34] In general, impactors provide sizing information in the size range from 0.3 to 20 μm diameter, and collect from 10 to 100 mg of dust, depending on the size distribution of the dust, its density, and whether a cyclone precutter (a cyclone operating upstream of the impactor) is used. The mechanism by which a cascade impactor operates is illustrated in Figure 12. In each stage of an impactor, the gas stream passes through an orifice and forms a jet which is directed toward an impaction plate. For each stage there is a characteristic particle diameter which has a 50% probability of impaction. This characteristic diameter is called the D_{50} of the stage. Although single jets are shown in Figure 12 for illustrative purposes, commercial impactors may have from one to several hundred jets in a stage. Typically, an impactor has five to ten stages.

The particle collection efficiency of a particular impactor jet-plate combination is determined by the properties of the aerosol, such as the particle shape and density, and the viscosity

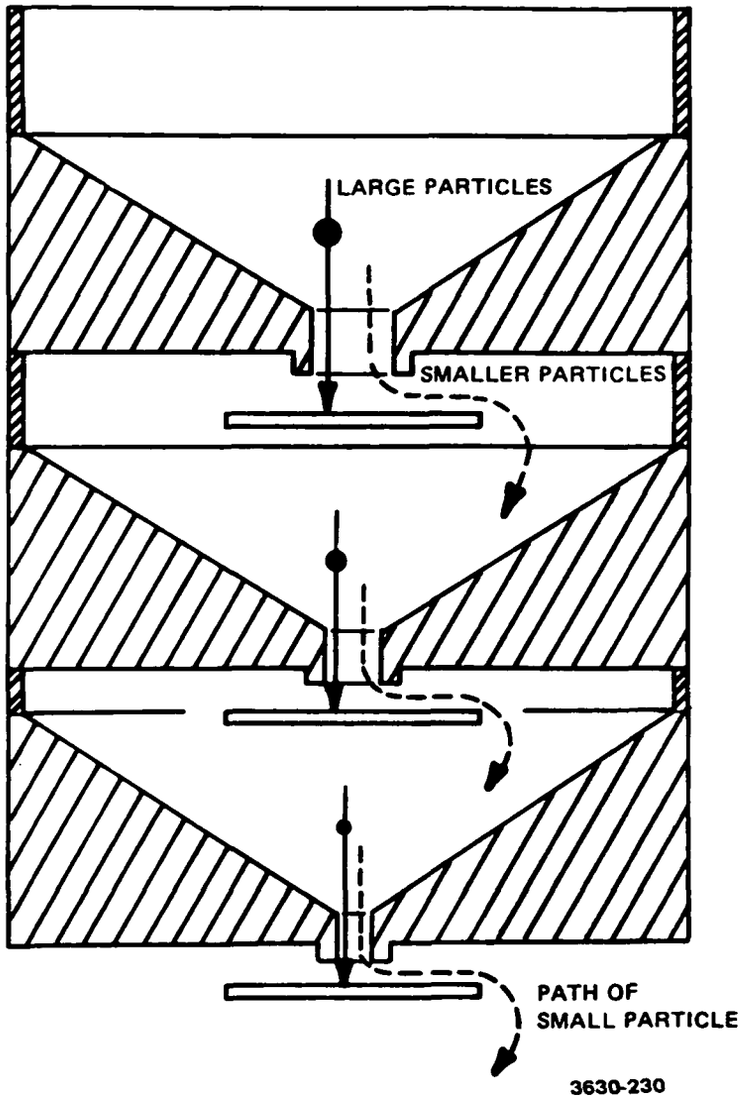


Figure 12. Schematic diagram, operation of cascade impactor.

of the gas; and by the design of the impactor stage, that is the shape of the jet, the diameter of the jet, and the jet-to-plate spacing.[35,36,37,38,39] There is also a slight dependence on the type of collection surface used (glass fiber, grease, metal, etc.).[40,41,42]

Table IV lists six commercially available cascade impactors that are designed for instack use, and schematics of five of them are shown in Figure 13.

The impactors are all constructed of stainless steel for corrosion resistance. All of the impactors have round jets, except the Sierra Model 226, which is a radial slit design, and all have stages with multiple jets, except the Brink. It is necessary to operate the impactors at a constant flow rate during a test so that the D_{50}'s will remain constant. The impactor flow rate is chosen, within a fairly narrow allowable range, to give a certain sampling velocity at the nozzle inlet. Streamlined nozzles of different diameters are provided to allow the sample to be taken at a velocity equal to that of the gas stream.

Since the impaction plates weigh a gram or more, and the typical mass collected on a plate during a test is on the order of 1-10 mg, it is often necessary to place a light weight collection substrate over the impaction plate to reduce the tare. These substrates are usually glass fiber filter material or greased aluminum foil. A second function of the substrates is to reduce particle bounce.

Cushing, et al. have done extensive calibration studies of the commercial, instack, cascade impactors.[41] Figure 14 shows results from calibration of the Andersen Mark III impactor that are typical of the performance of the other types as well. The decrease in collection efficiency for large particles represents bounce and can introduce serious errors in the calculated particle-size distribution.

TABLE IV

COMMERCIAL CASCADE IMPACTOR SAMPLING SYSTEMS

Name	Nominal Flow rate (cm^3/sec)	Substrates	Manufacturer
Andersen Stack Sampler (Precollection Cyclone Avail.)	236	Glass Fiber (Available from manufacturer)	Andersen 2000, Inc. P.O. Box 20769 Atlanta, GA 30320
Univ. of Washington Mark III Source Test Cascade Impactor (Precollection Cyclone Avail.)	236	Stainless Steel Inserts, Glass Fiber, Grease	Pollution Control System Corp. 321 Evergreen Bldg. Renton, WA 98055
Univ. of Washington Mark V	100	Stainless Steel Inserts, Glass Fiber, Grease	Pollution Control System Corp. 321 Evergreen Bldg. Renton, WA 98055
Brink Cascade Impactor (Precollection Cyclone Avail.)	14.2	Glass Fiber, Aluminum, Grease	Zoltek Corp. 68 Worthington Drive St. Louis, MO 63043
Sierra Source Cascade Impactor - Model 226 (Precollection Cyclone Avail.)	118	Glass Fiber (Available from manufacturer)	Sierra Instruments, Inc. P.O. Box 909 Village Square Carmel Valley, CA 93924
MRI Inertial Cascade Impactor	236	Stainless Steel, Alumnnum, Mylar, Teflon. Optional: Gold, Silver, Nickel	Meteorology Research, Inc. Box 637 Altadena, CA 91001

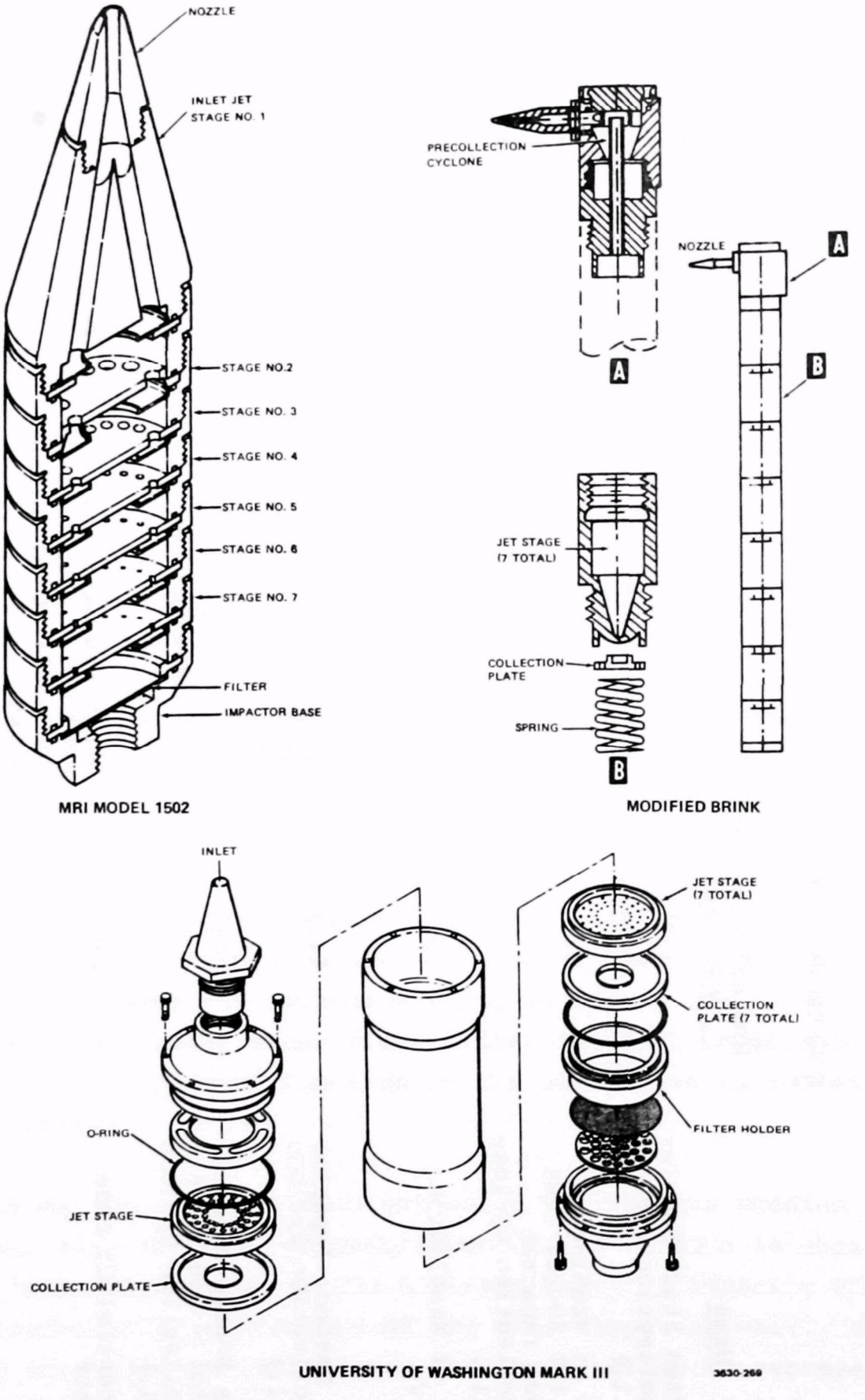


Figure 13. Schematics of five commercial cascade impactors (Sheet 1 of 2).

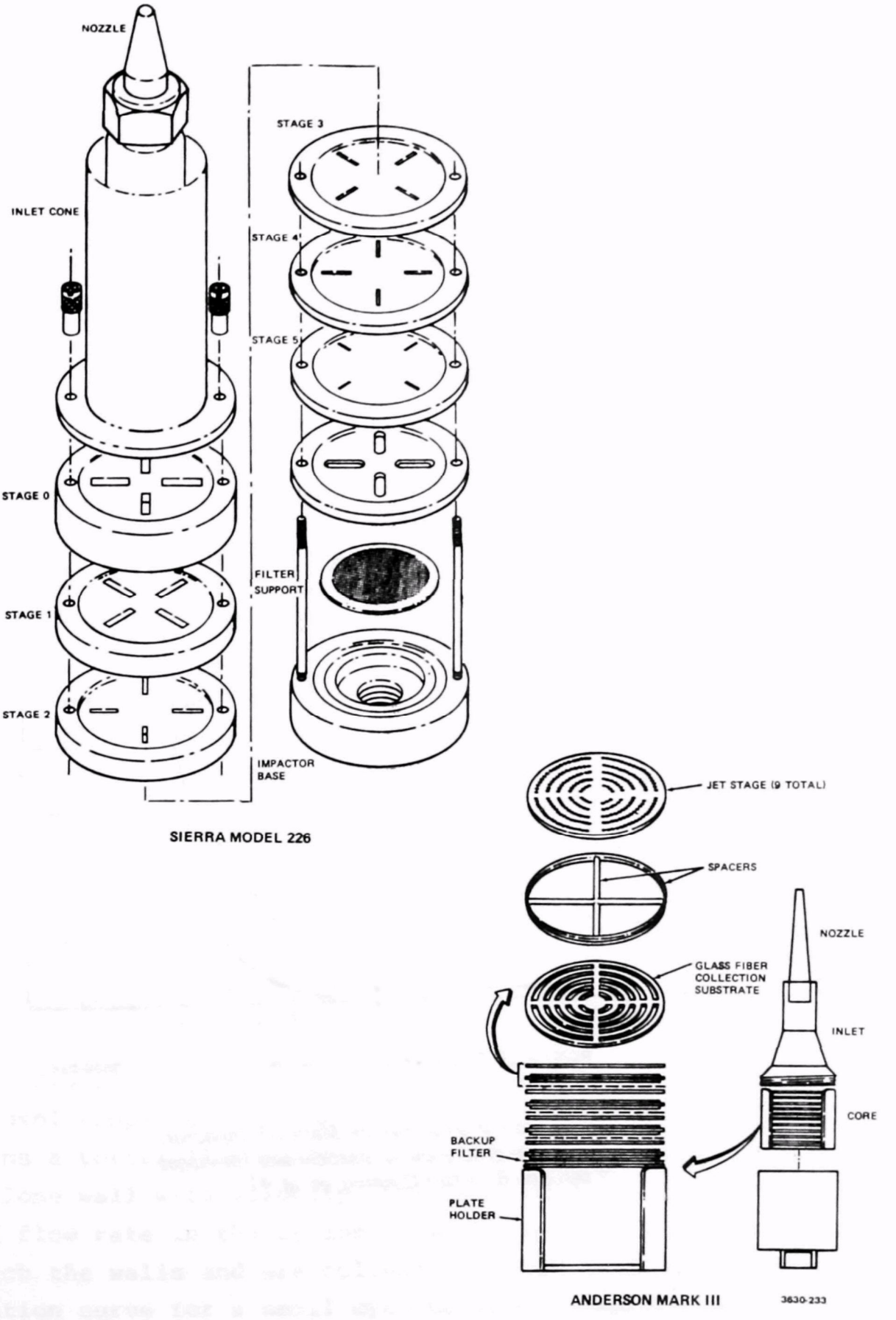


Figure 13. Schematics of five commercial cascade impactors (Sheet 2 of 2).

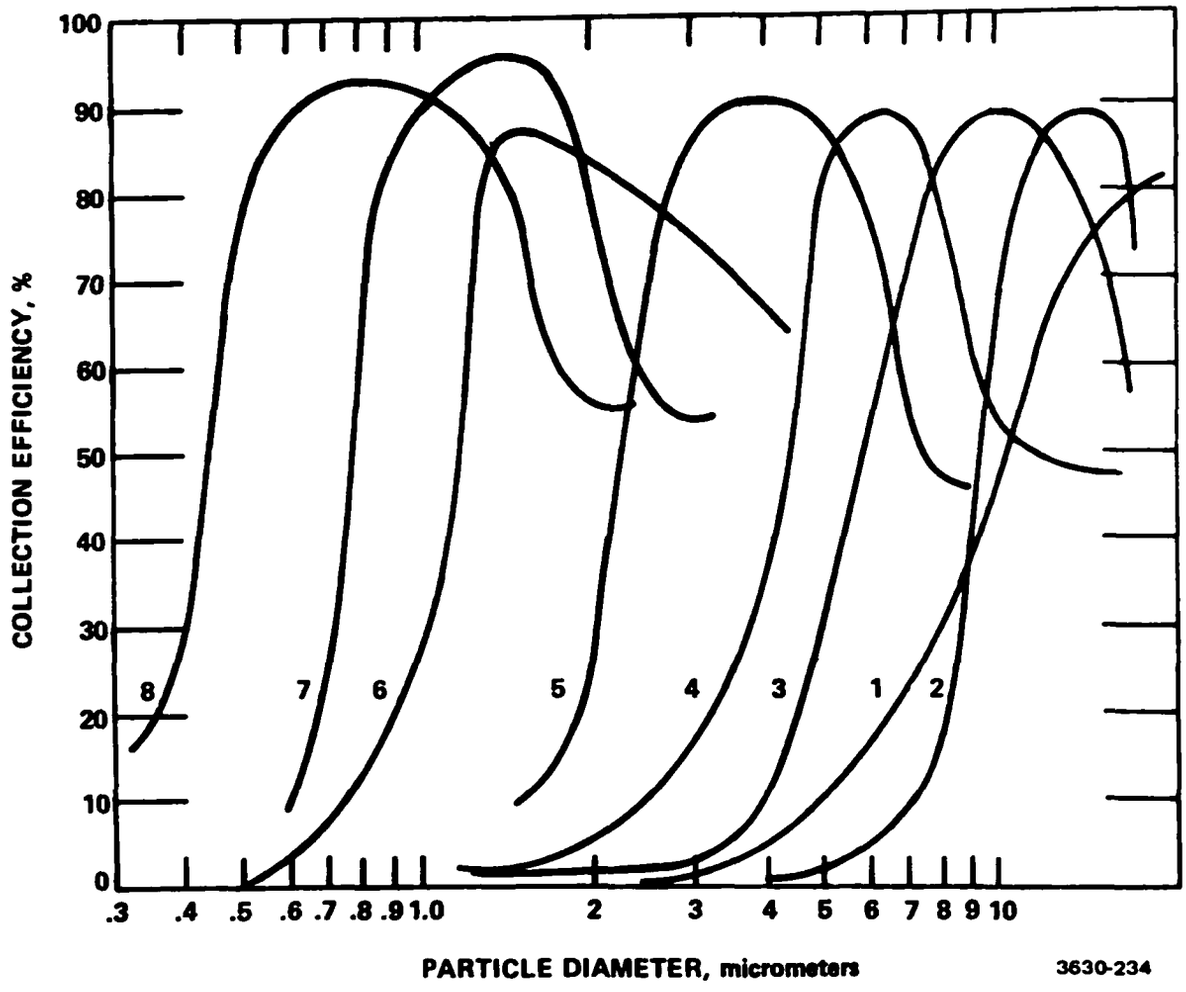


Figure 14. Calibration of an Anderson Mark III impactor. Collection efficiency vs. particle size for stages 1 through 8. After Cushing, et al.[41]

There has not been an extensive evaluation of cascade impactors under field conditions, although some preliminary work was reported by McCain, et al.[43] It is difficult to judge from existing data exactly how accurate impactors are, or how well the data taken by different groups or with different impactors will correlate. Problems that are known to exist in the application of impactors in the field are: substrate instability,[44,45] the presence of charge on the aerosol particles,[46] particle bounce,[40,47] and mechanical problems in the operation of the impactor systems.

In the past, the reduction of data from an extensive field test has been excessively tedious and time consuming. However, a computer program is now available that decreases the effort required to reduce and analyze impactor data by approximately a factor of five.[48]

Cyclones--Cyclones have been used for many years as devices for cleaning dusty air and also to separate respirable and nonrespirable dusts in personal exposure monitors. Strauss[49] has reviewed in detail the theory, design, and performance of industrial cyclones, while Lippmann and Chan have performed several experimental/theoretical studies of the small cyclones used as personal exposure monitors.[50,51] In general, it can be said that the existing theories are not accurate enough to design cyclones for particle sizing, and thus such designs must be developed empirically.

Figure 15 illustrates a typical reverse flow cyclone. The aerosol sample enters the cyclone through a tangential inlet and forms a vortex flow pattern. Particles move outward toward the cyclone wall with velocity that is determined by the geometry and flow rate in the cyclone, and by their size. Large particles reach the walls and are collected. Figure 16 compares the calibration curve for a small cyclone with a typical impactor calibration curve. The cyclone can be seen to perform almost as well

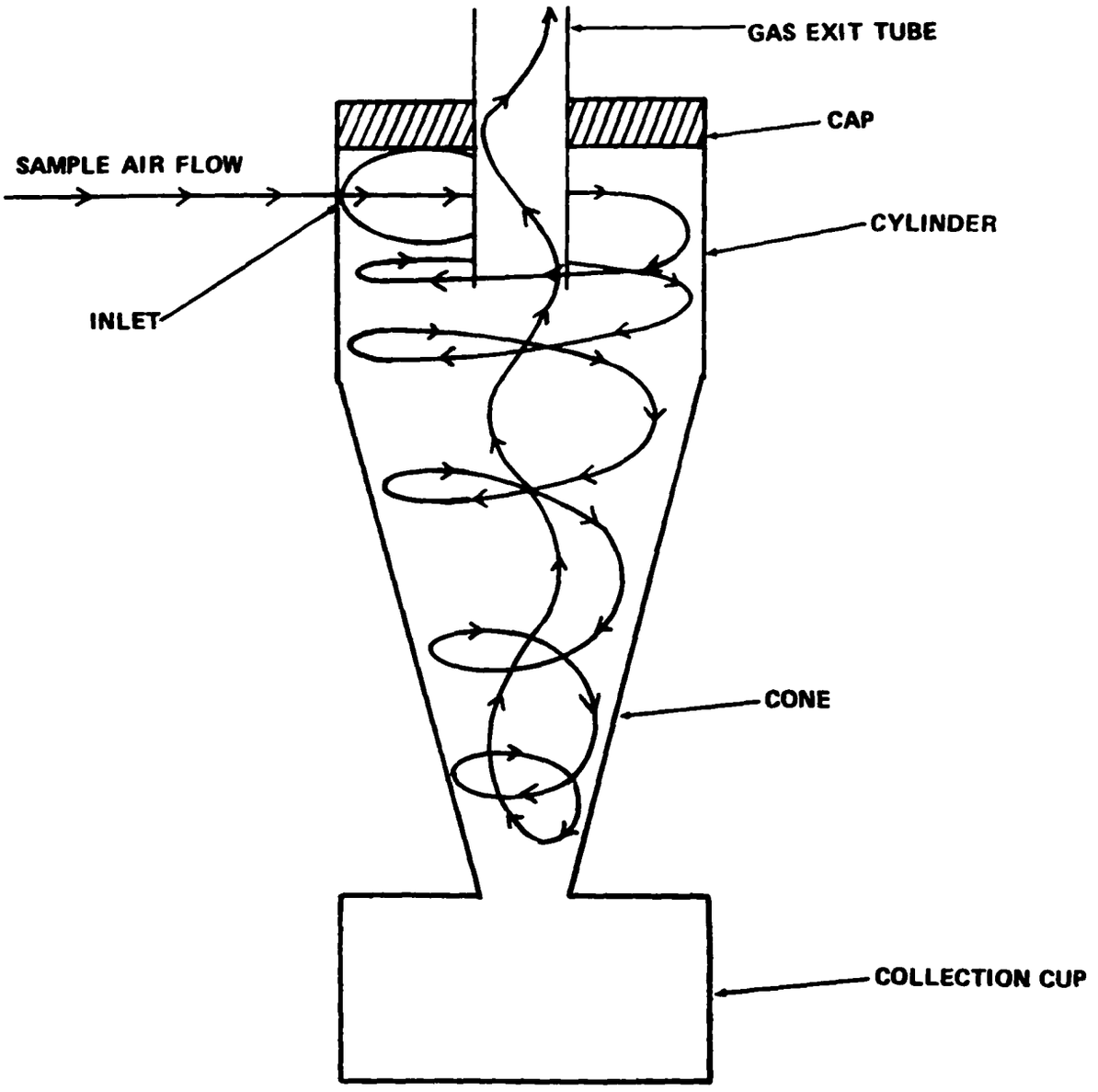


Figure 15. Hypothetical flow through a cyclone of conventional design.

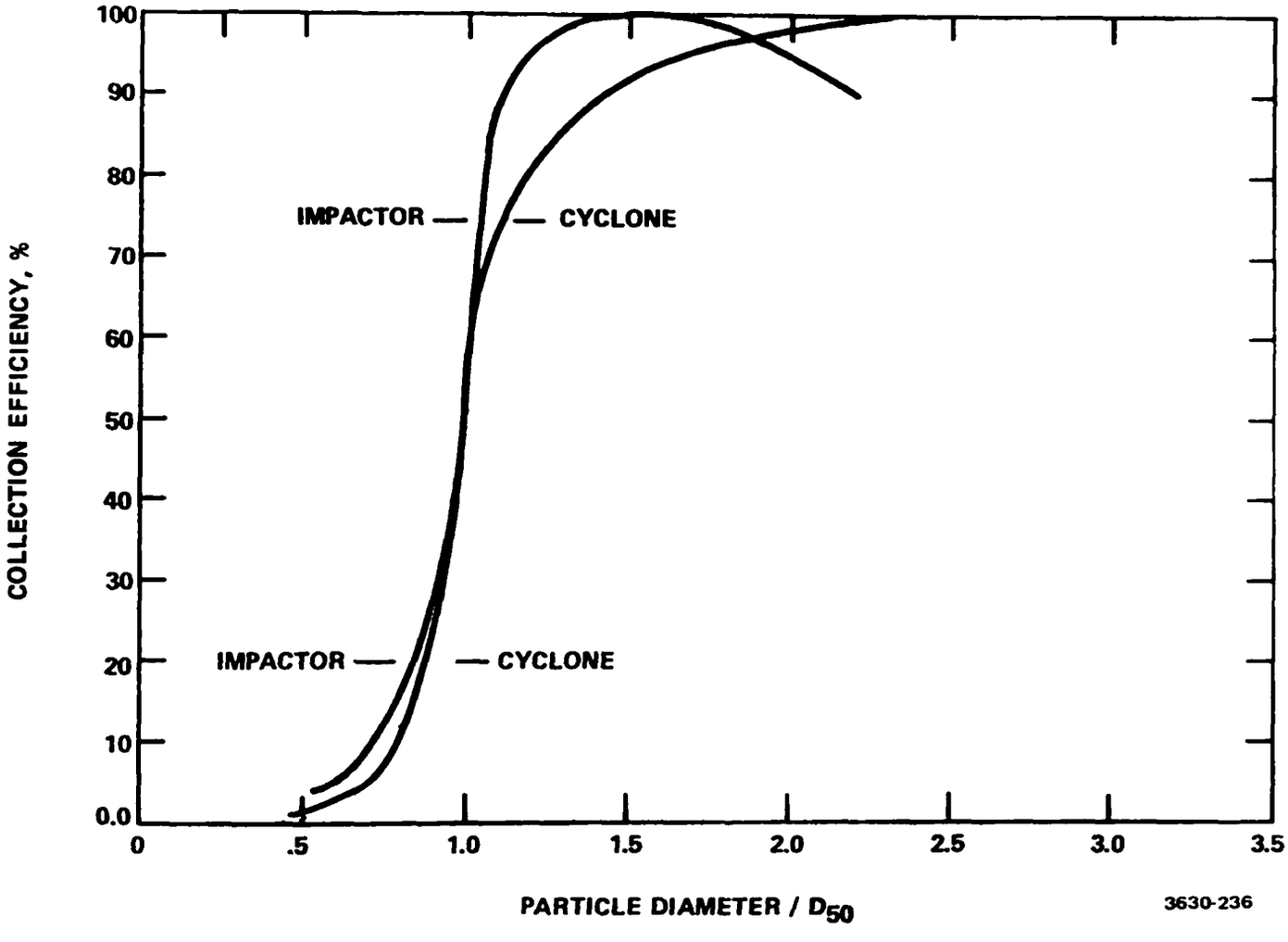


Figure 16. Comparison of cascade impactor stage with cyclone collection efficiency curve.

as the impactor, and the problem of large particle bounce and reentrainment is absent.

A series of cyclones with progressively decreasing D_{50}'s can be used instead of impactors to obtain particle size distributions, with the advantages that larger samples are acquired and that particle bounce is not a problem. Longer sampling times are possible with cyclones because of their large dust capacity (the collection cup may hold several grams of dust without affecting the performance of the cyclone). This is an advantage for sampling very dusty streams because it allows longer run times.

Southern Research Institute, under EPA sponsorship, has designed and built a prototype three-stage series cyclone system for in-stack use.[52] A sketch of this system is shown in Figure 17. It is designed to operate at 472 cm^3/sec (1 ft^3/min). The D_{50}'s for these cyclones are 3.0, 1.6, and 0.6 micrometer aerodynamic at 21°C. A 47 mm Gelman filter holder, (Gelman Instrument Co., 600 South Wagner Road, Ann Arbor, MI 48106), is used as a back up filter after the last cyclone. This series cyclone system was designed for in-stack use and requires a 15 cm diameter sampling port.

Figure 18 illustrates a second generation EPA/Southern Research series cyclone system now under development which contains five cyclones and a back up filter and will fit through 10 cm diameter ports. Prototypes of anodized aluminum, titanium, (for in-stack evaluation), and Hastelloy (for high temperature and pressure sampling) have been constructed and are under evaluation. Figure 19 contains laboratory calibrations data for the five cyclone prototype system. The D_{50}'s at the test conditions are 0.32, 0.65, 1.4, 1.6, 2.1, and 5.4 micrometers.[53]

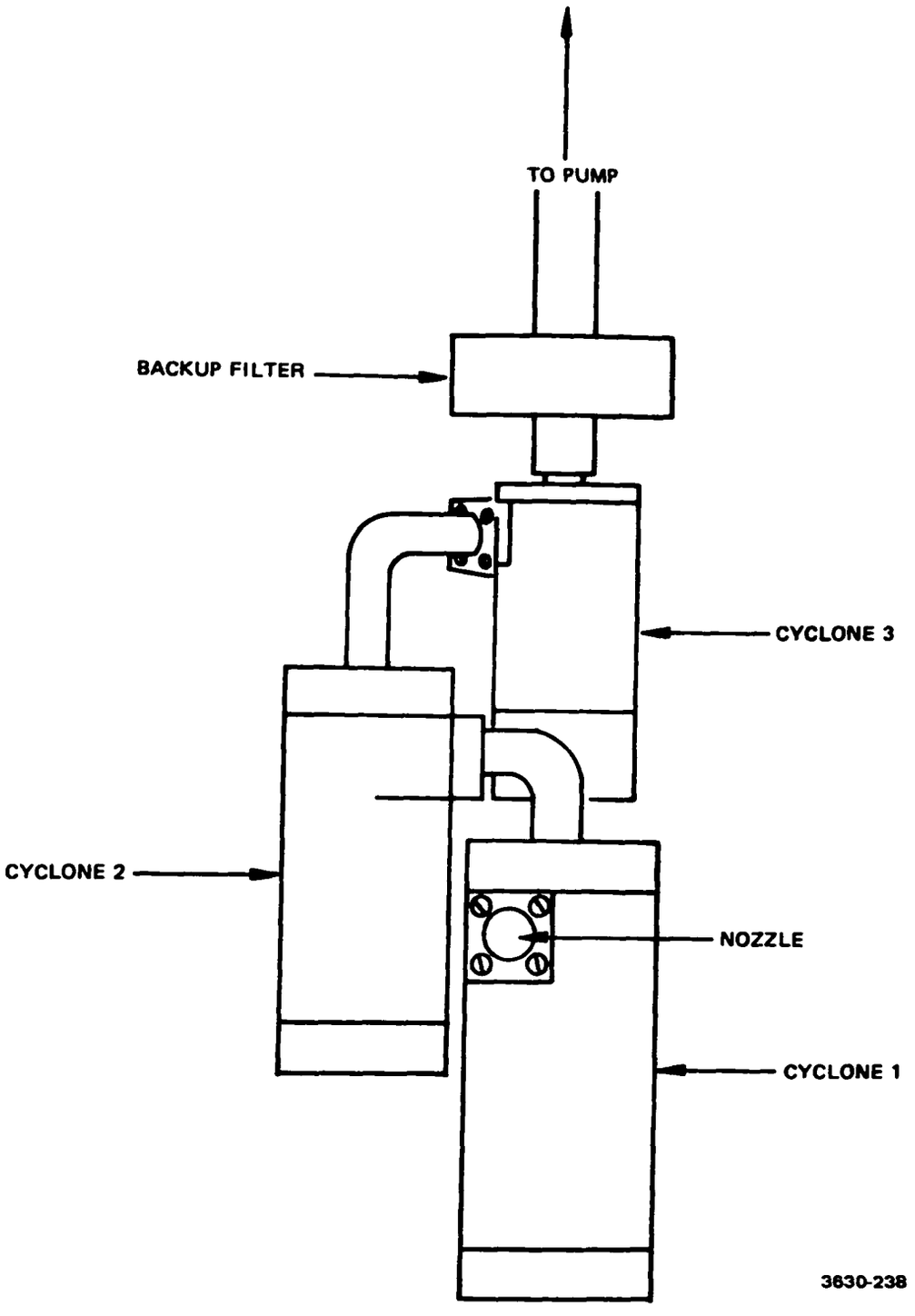


Figure 17. Schematic of the Southern Research Institute Three Series Cyclone System.

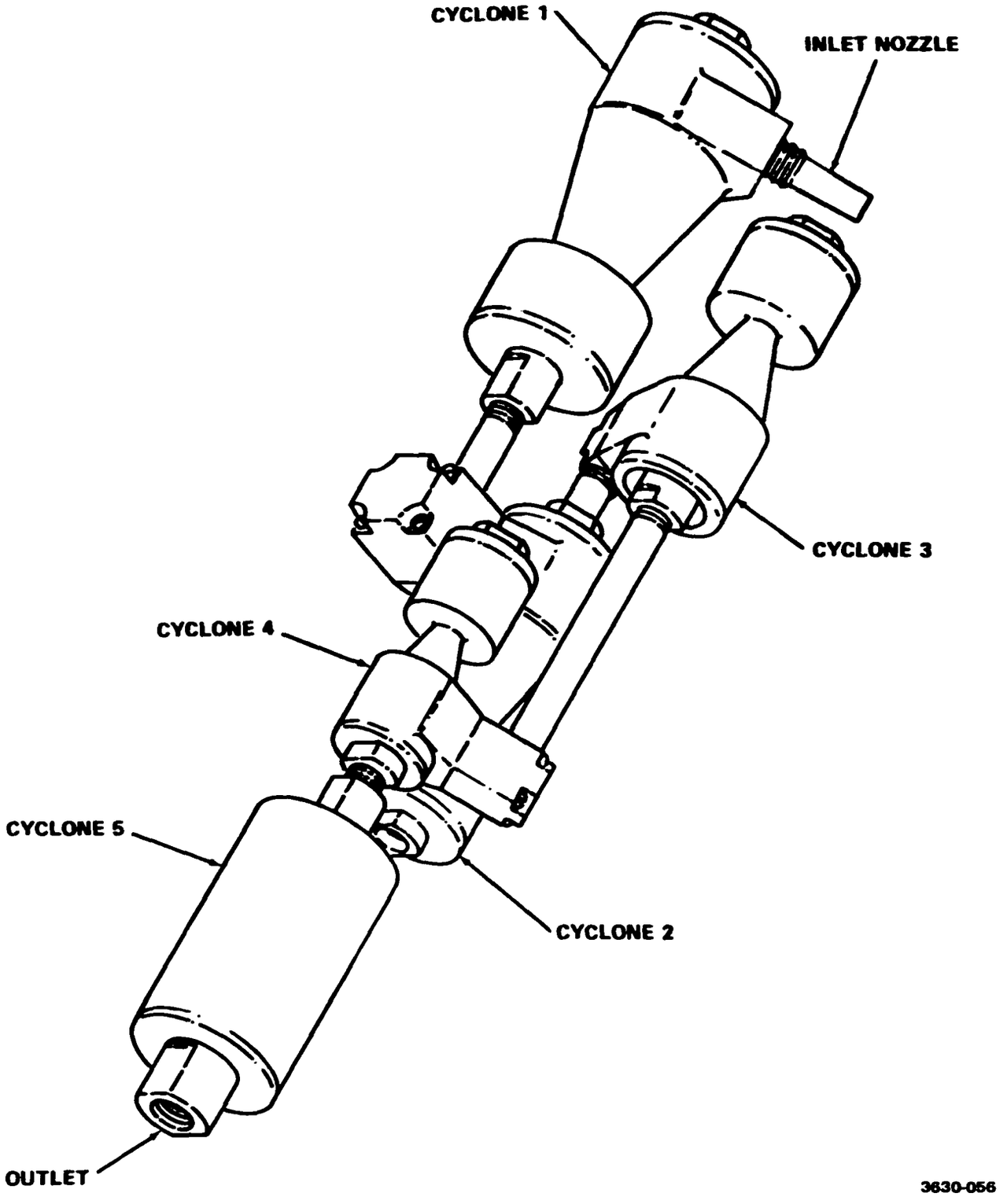


Figure 18. Environmental Protection Agency-Southern Research Institute Five-Stage Cyclone.

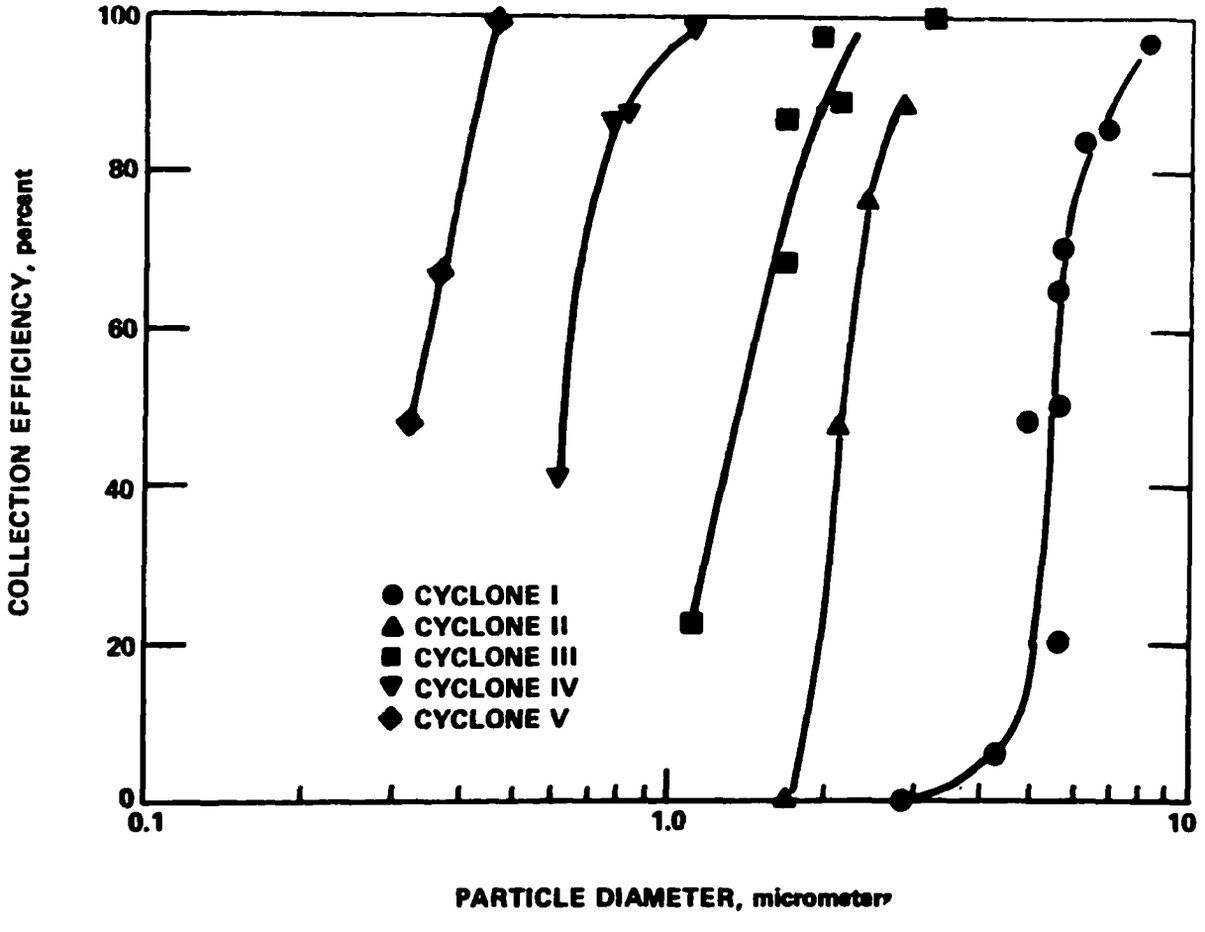


Figure 19. Collection efficiency of the EPA-S.R.I. Cyclones at a flow rate of 28.3 ℓ/min, a temperature of 25°C, and for a particle density of 1.00 gm/cm³.

The Acurex-Aerotherm Source Assessment Sampling System (SASS) incorporates three cyclones and a back-up filter.[54] Shown schematically in Figure 20, the SASS is designed to be operated at a flow rate of 3065 cm^3/sec (6.5 ft^3/min) with nominal cyclone D_{50}'s of 10, 3, and 1 micrometer aerodynamic diameter at a gas temperature of 205°C. The cyclones, which are too large for in situ sampling, are heated in an oven to keep the air stream from the heated extractive probe at stack temperature or above the dew point until the particulate is collected. Besides providing particle size distribution information, the cyclones collect gram quantities of dust (due to the high flow rate) for chemical and biological analyses. The SASS train is available from Acurex-Aerotherm, Inc., 485 Clyde Ave., Mountain View, California 94042.

Small cyclone systems appear to be practical alternatives to cascade impactors as instruments for measuring particle size distributions in process streams under conditions where it is appropriate to sample for longer periods and to obtain larger samples. Additional investigations are underway to obtain a more detailed understanding of cyclones used for sampling.

Optical Particle Counters--

Figure 21 is a schematic illustrating the principle of operation for optical particle counters. A dilute aerosol stream intersects the focus of a light beam to form an optical "view volume." The photodetector is located so that no light reaches its sensitive cathode except that scattered by particles in the view volume. Thus, each particle that scatters light with enough intensity will generate a current pulse at the photodetector, and the amplitude of the pulse can be related to the particle diameter. Optical particle counters yield real-time information on particle size and concentration.

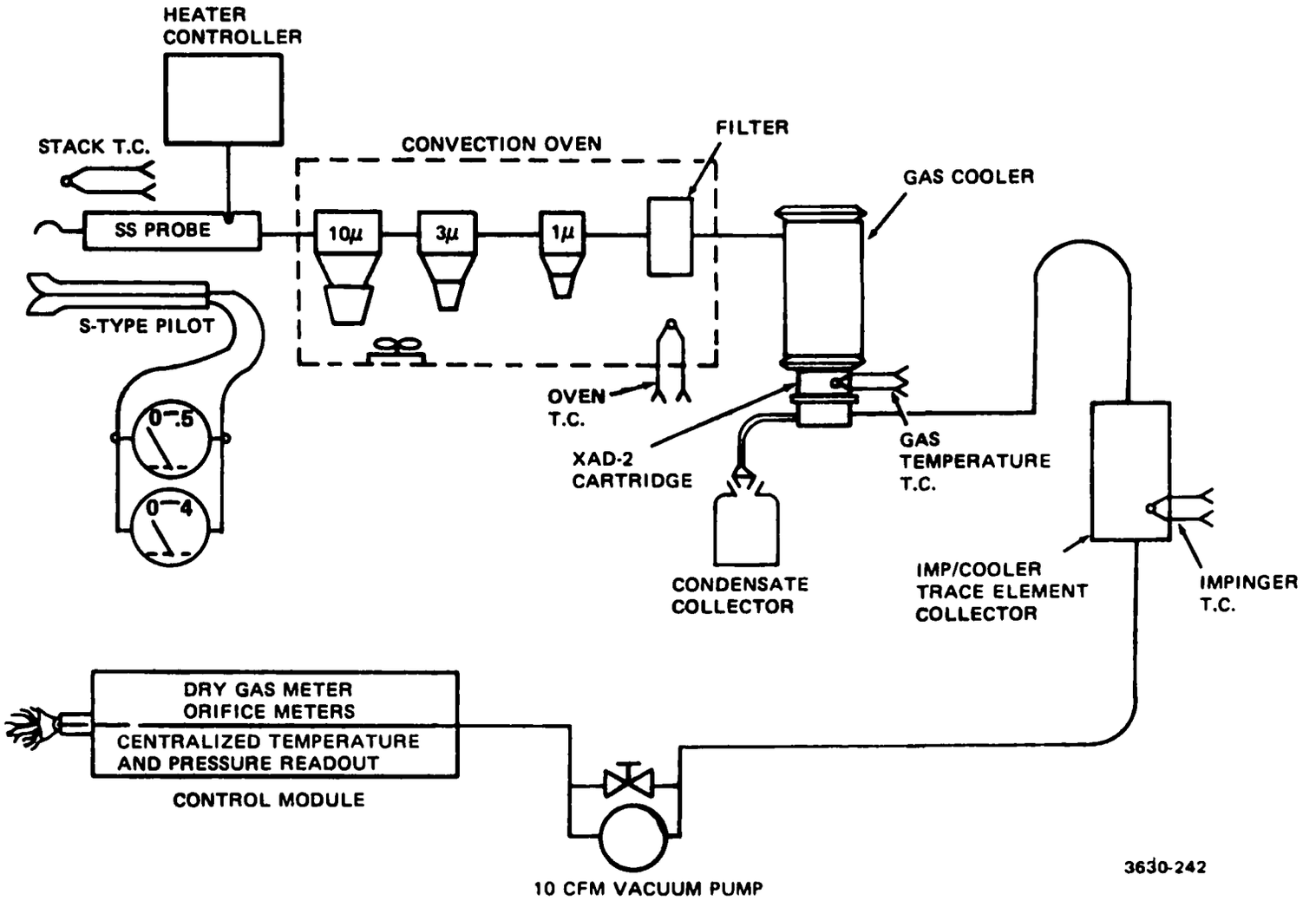


Figure 20. Schematic of the Acurex-Aerotherm Source Assessment Sampling System (SASS).

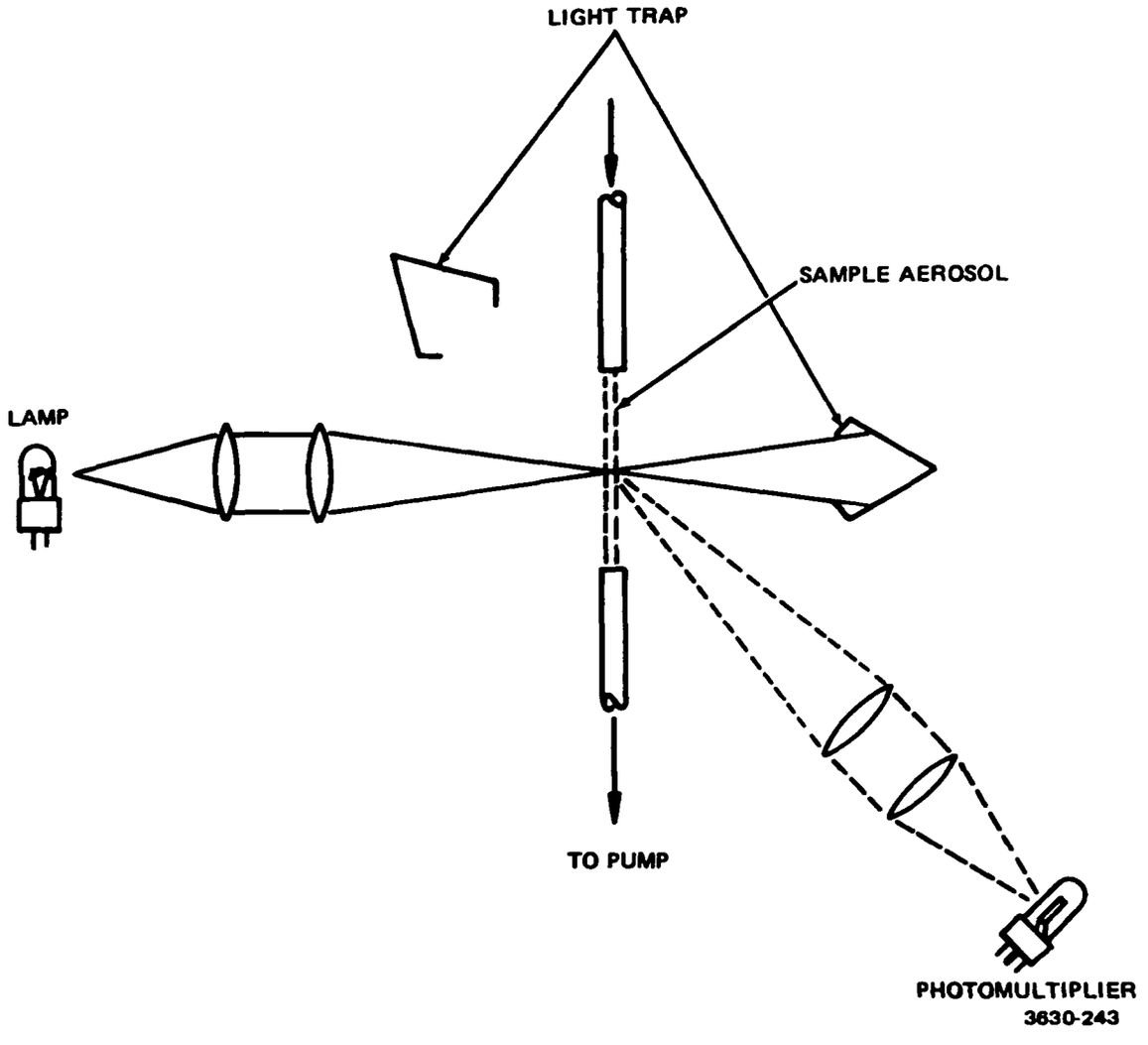


Figure 21. Schematic of an optical single particle counter.

Figure 22 illustrates some of the optical configurations that are found in commercial particle counters. The pertinent geometric and operating constants of the counters are summarized in Table V.

The commercial optical counters that are available now were designed for laboratory work and have concentration limits of a few hundred particles per cubic centimeter. The lower size limit is nominally about 0.3 μm diameter. For use in studies of industrial aerosols, dilution of the sample is required and the useful upper limit in particle size has been limited by losses in the dilution system to about 2.0 μm diameter.[55] In addition, the particle diameter that is measured is not aerodynamic, and some assumptions must be made in order to compare optical with aerodynamic data. Nevertheless, the ability to obtain real-time information can sometimes be very important and the special problems in sampling with optical counters may be justified.

Diffusion Batteries with Condensation Nuclei Counters--

The classical technique for measuring the size distribution of submicron particles employs the relationship between particle diffusivity and diameter. In a diffusional sizing system, the test aerosol is drawn, under conditions of laminar flow, through a number of narrow, rectangular channels, a cluster of small bore tubes, or a series of small mesh screens (diffusion batteries). For a given particle diameter and diffusion battery geometry, it is possible to predict the rate at which particles are lost to the walls by diffusion, the rate being higher for smaller particles. The total number of particles penetrating the diffusion battery is measured under several test conditions where the main adjustable parameter is the aerosol retention time, and the particle-size distribution is calculated by means of suitable mathematical deconvolution techniques. Figure 23 illustrates the geometry of a rectangular channel diffusion battery, and Figure 24 a screen-type diffusion battery.

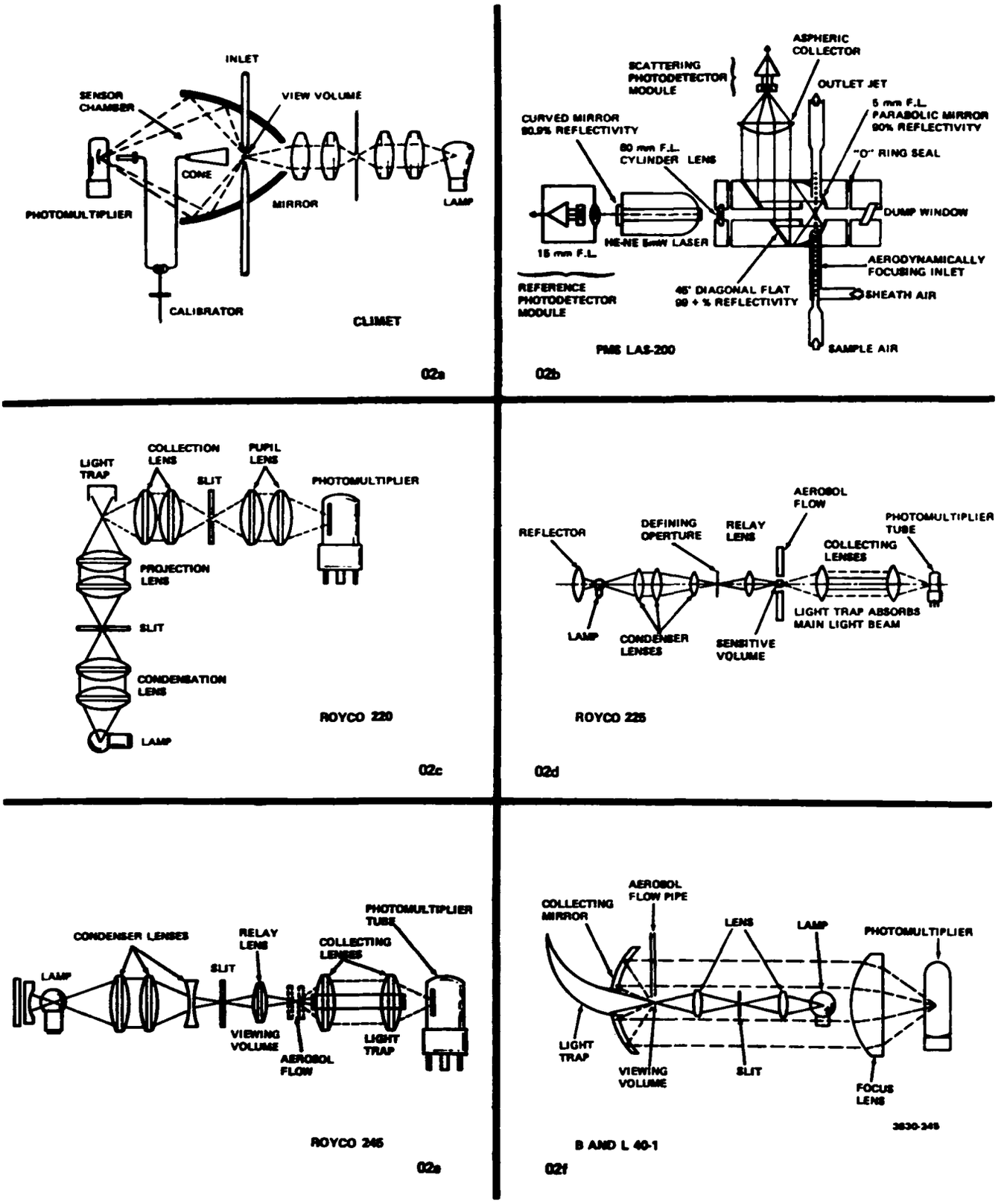


Figure 22. Optical configurations for six commercial particle counters.

TABLE V.

CHARACTERISTICS OF COMMERCIAL, OPTICAL, PARTICLE COUNTERS

	Illuminating Cone Half Angle, γ	Light Trap Half Angle, α	Collecting Aperture Half Angle, β	Inclination Between Illuminating And Collecting Cone Axis, ψ	Viewing Volume	Sampling Rate
Bausch & Lomb Model 40-1 820 Linden Avel Rochester, NY 14625	13°	33°	53°	0°	0.5 mm^3	170 cm^3/min
Climet Models 201, 208 Climet Inst. Co. 1620 W. Colton Ave. Redlands, CA 92373	15	35	90	0	0.5	7,080
Climet Model 150	12	18	28	0	0.4	472
Royco Model 218 Royco Inst. 41 Jefferson Dr. Menlo Park, CA 94025	5	11	30	0	0.25	283
Royco Model 220	24	-	24	90	2.63	2,830
Royco Model 245	5	16	25	0	4.0	28,300
Royco Model 225	5	7	25	0	2.0	283 or 2,830
Tech Ecology Model 200 Tech Ecology, Inc. 645 N. Mary Ave. Sunnyvalle, CA 94086	5	8	20	0	0.46	283
Tech Ecology Model 208	5	10	20	0	2.5	2,830
Particle Measurement Systems	0.5	35	120	0	0.003	120 or 1,200
*Model LAS-200 Particle Measuring Systems 1855 S. 57th Ct. Boulder, CO 80301						

*632.8 mm laser illum., all others are white light.

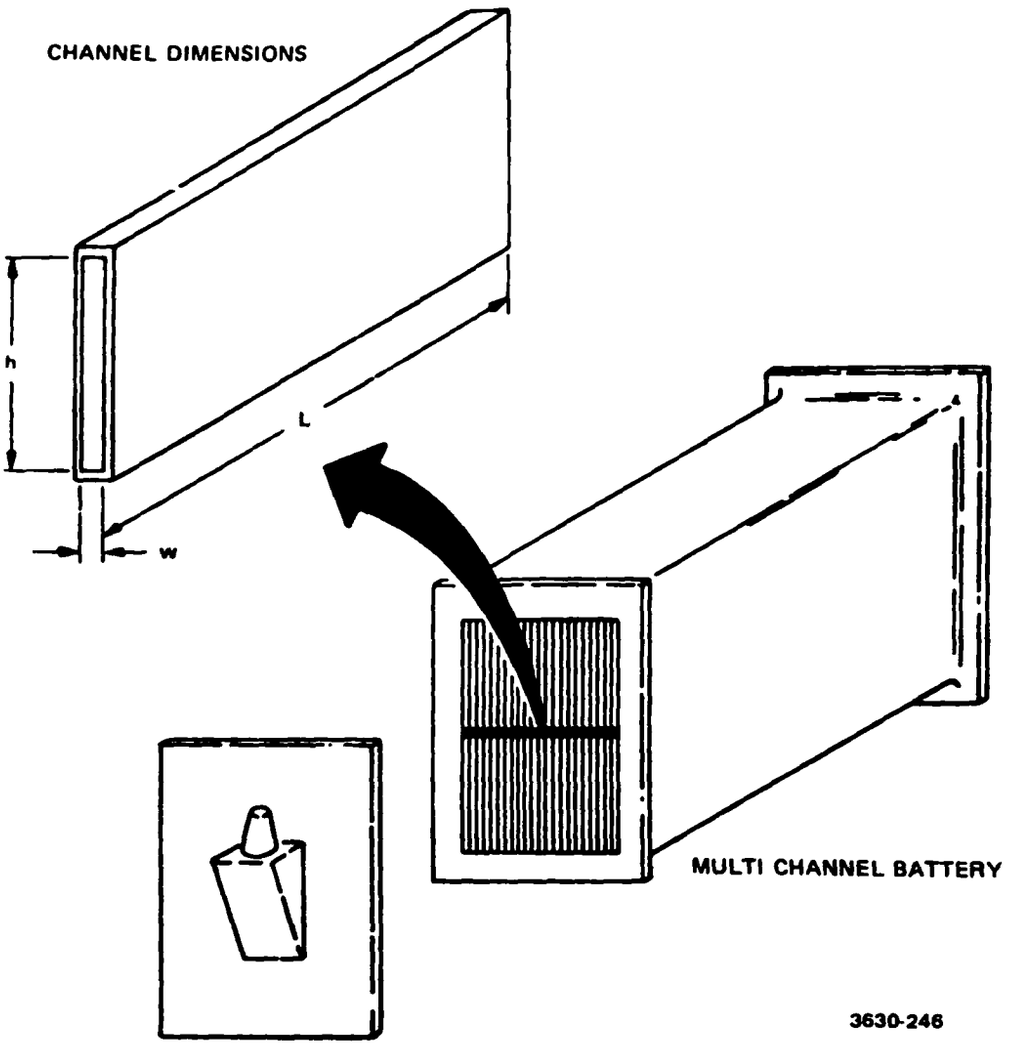


Figure 23. A rectangular channel diffusion battery.

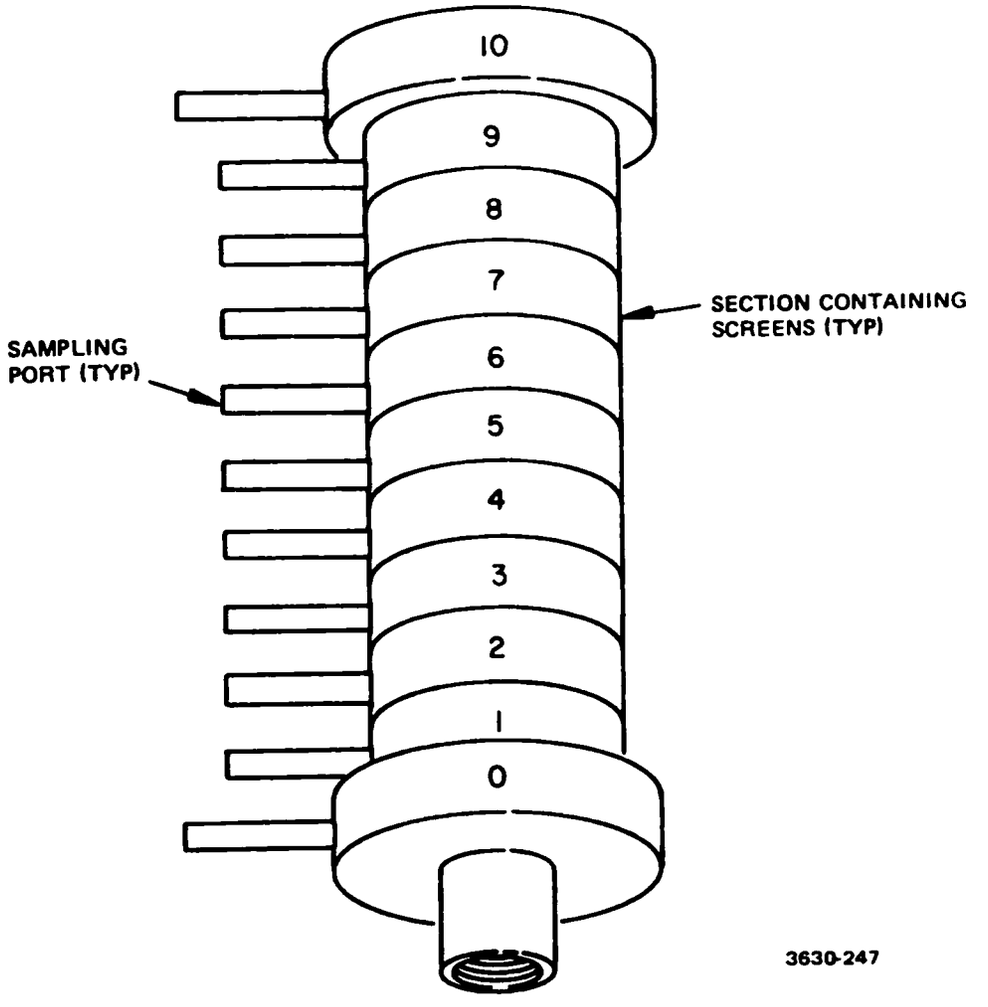


Figure 24. Screen type diffusion battery. The battery is 21 cm long, 4 cm in diameter, and contains 55, 635 mesh stainless steel screens. After Sinclair. [56]

Condensation nuclei (CN) counters function on the principle that particles act as nuclei for the condensation of water or other condensable vapors in a supersaturated environment. This process is used to detect and count particles in the 0.002 to 0.3 micron range (often referred to as condensation or Aitken nuclei). In condensation nuclei detectors, a sample is withdrawn from the gas stream, humidified, and brought to a supersaturated condition by reducing the pressure. In this supersaturated condition, condensation will be initiated on all particles larger than a certain critical size and will continue as long as the sample is supersaturated. This condensation process forms a homogeneous aerosol, predominantly composed of the condensed vapor containing one drop for each original particle whose size was greater than the critical size appropriate to the degree of supersaturation obtained; a greater degree of supersaturation is used to initiate growth on smaller particles. The number of particles that are formed is estimated from the light scattering properties of the final aerosol. Figure 25, after Haberl and Fusco, illustrates the condensation nuclei counter operating principle.[57]

Four models of CN counters are now available commercially. Two automatic, or motorized, types are the General Electric Model CNC-2 (General Electric-Ordnance Systems, Electronics Systems Division, Pittsfield, MA 01201) and the Environment-One Model Rich 100 (Environment-One Corporation, Schenectady, NY 12301). Small, manually operated, CN counters are also available from Gardner Associates (Gardner Associates, Schenectady, NY 12301), and Environment-One.

Thermosystems, Inc. (Thermosystems, Inc., St. Paul, MN 55113) now manufactures and sells screen-type diffusion batteries of Sinclair design (Figure 24). These diffusion batteries are 21 cm long, approximately 4 cm in diameter, weigh 0.9 kg, and contain 55 stainless steel screens of 635 mesh.

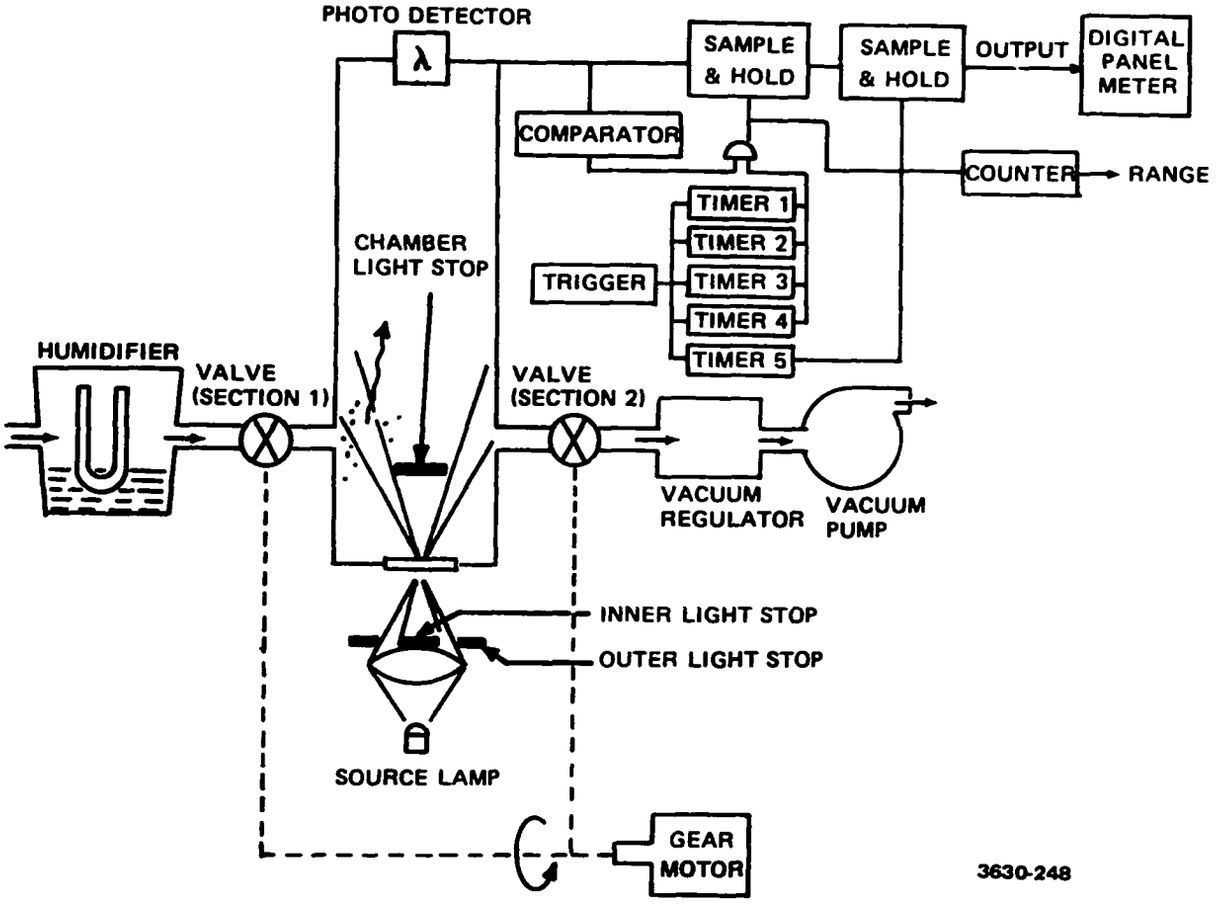


Figure 25. Diagram of a condensation nuclei counter. After Haberl and Fusco. [57]

Figure 26 is a schematic that illustrates an experimental setup for measuring particle-size distributions by diffusional means, and Figure 27 shows penetration curves for four operating configurations. Because of the long retention time required for removal of particles by diffusion, measurements with diffusion batteries and CN counters are very time consuming. With the system described by Ragland, et al., for example, approximately two hours are required to measure a particle-size distribution from 0.01 to 0.2 μm.[58] Obviously, this method is best applied to stable aerosol streams. It is possible that the new, smaller diffusion batteries will allow much shorter sampling times, but pulsations in flow may pose a serious problem for the low volume geometries.

Electrical Mobility--

Figure 28 illustrates the relationship between the diameter and electrical mobility of small aerosol particles. If particles larger than those of minimum mobility are removed from the sample, the remaining particles exhibit a monotonically decreasing mobility with increasing diameter. Several aerosol spectrometers, or mobility analyzers, have been demonstrated that employ the diameter-mobility relationship to classify particles according to their size,[60,61,62,63] and Figure 29 illustrates the principle on which these devices operate. Particles are charged under conditions of homogeneous electric field and ion concentration, and then passed into the spectrometer. Clean air flows down the length of the device and a transverse electric field is applied. From a knowledge of the system geometry and operating conditions, the mobility is derived for any position of deposition on the grounded electrode. The particle diameter is then readily calculated from a knowledge of the electric charge and mobility.

Difficulties with mobility analyzers are associated primarily with charging the particles to a known value with a minimum of

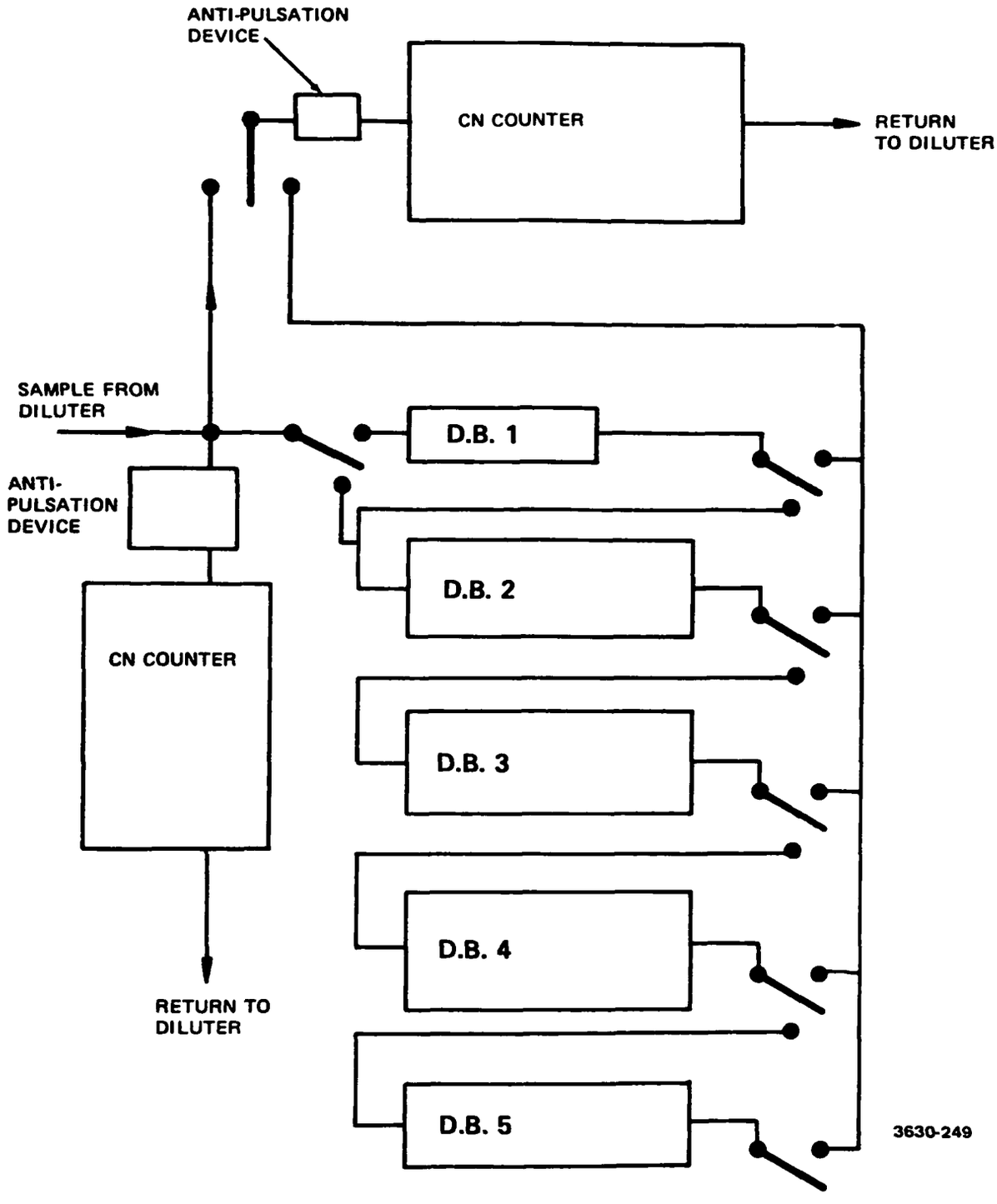


Figure 26. Diffusion battery and condensation nuclei counter layout for fine particle sizing.[19]

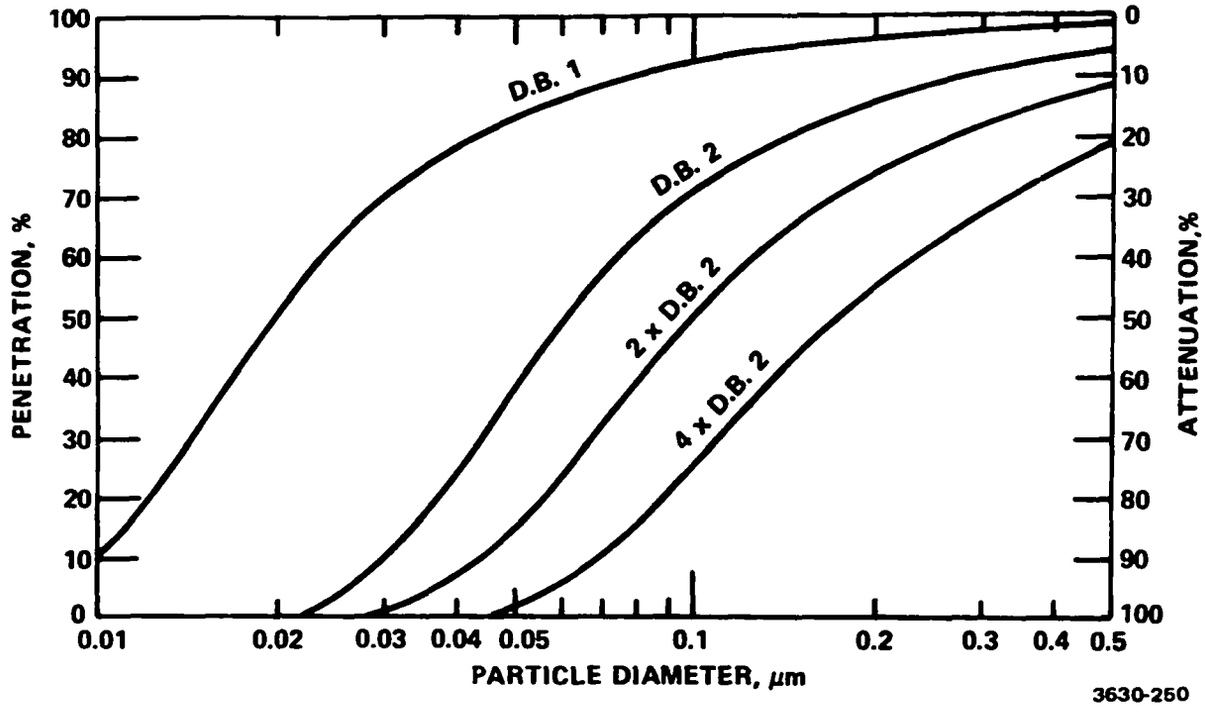


Figure 27. Theoretical parallel plate diffusion battery penetration curves.

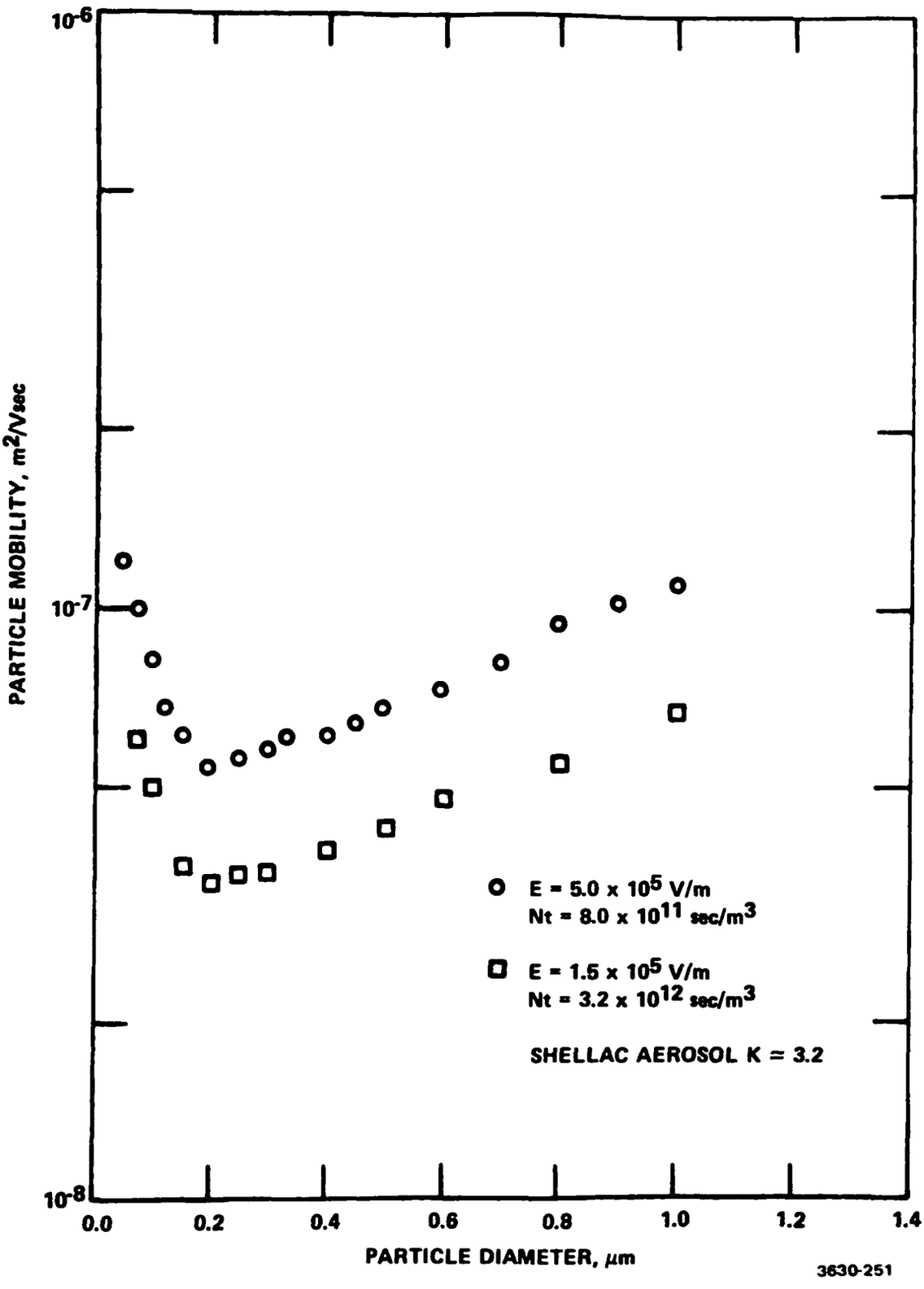


Figure 28. Particle mobility as a function of diameter for shellac aerosol particles charged in a positive ion field (after Cochet and Trillat 59). K is the dielectric constant of the aerosol particles.

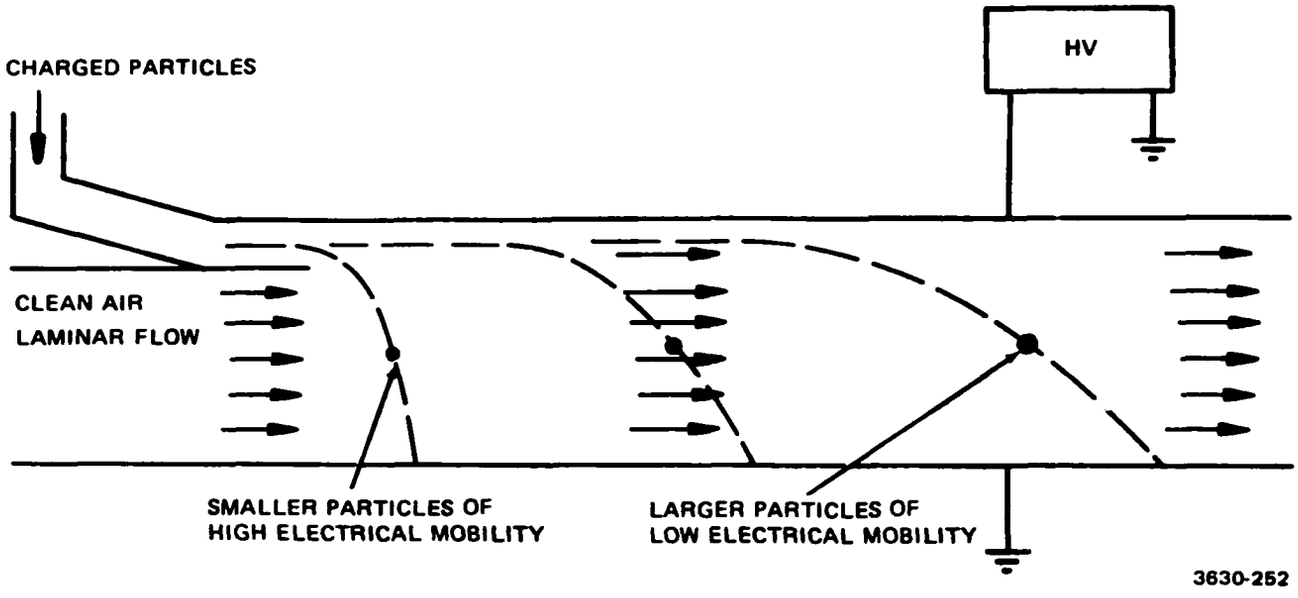


Figure 29. The electric mobility principle.

loss by precipitation and obtaining accurate analyses of the quantity of particles in each size range. The latter may be done gravimetrically,[60] optically,[61] or electrically.[62]

The concept described above has been used by Whitby, Liu, et al.,[64,65] at the University of Minnesota, to develop a series of models of the Electrical Aerosol Analyzer (EAA). A commercial version of the University of Minnesota devices is now marketed by Thermosystems, Inc., as the Model 3030 (Figure 30). The EAA is designed to measure the size distribution of particles in the range from 0.0032 to 1.0 μm diameter. The concentration range for best operation is 1 to 1000 $\mu g/m^3$, and thus dilution is required for most industrial gas aerosols.[66,67]

When the EAA is applied to fluctuating sources a peculiar problem arises. The instrument reading is cumulative, and it is impossible to tell whether variations in the reading reflect changes in the distribution of concentration of particles; hence, recordings that show rapid fluctuations in amplitude must be interpreted with great care. The lack of sensitivity can also be a problem at extremely clean sources.

The EAA requires only two minutes to perform a complete size distribution analysis, which generally makes it advantageous to use, especially on stable sources.

Laboratory Measurements

Measurements of the size distribution of particles that have been collected in the field and transported to a laboratory must be interpreted with great caution, if not skepticism. It is difficult to collect representative samples in the first place, and it is almost impossible to reconstruct the original size distribution under laboratory conditions. For example, one cannot distinguish from laboratory measurements whether or not some of the particle existed in the process gas stream as agglomerates of smaller particles. In spite of the limitations inherent in

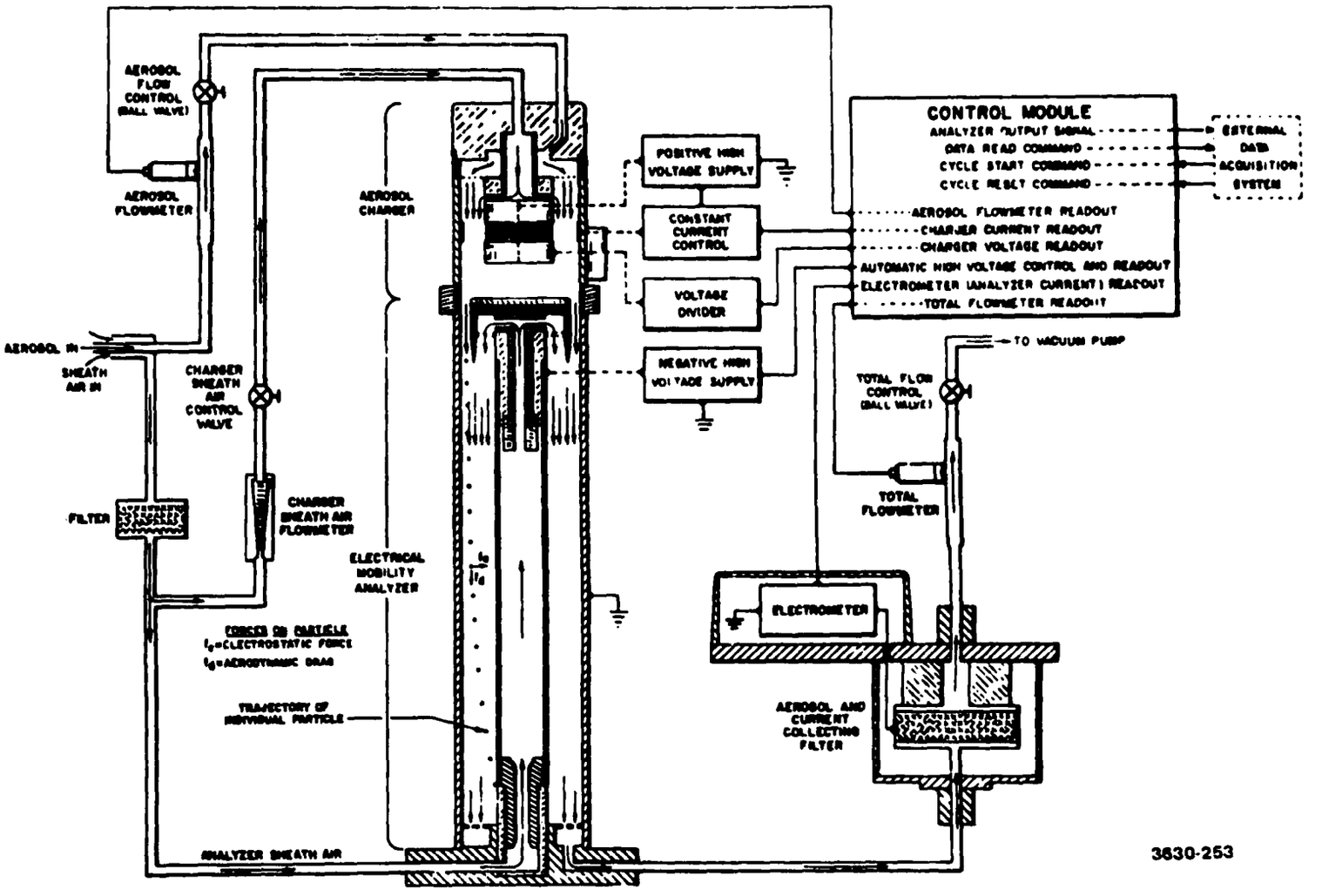


Figure 30. Schematic of the Thermosystems Model 3030 Electrical Aerosol Analyzer.

laboratory methods, they must be used in some instances to determine particle size and to segregate particles for determination of their composition or other properties of interest. This section contains a discussion of some of the "standard" techniques used for particle size analysis of dust samples.

Sedimentation and Elutriation--

Elutriation and sedimentation devices separate particles that are dispersed in a fluid according to their settling velocities due to gravity. Large particles in a quiescent aerosol will settle to the bottom region of the chamber more quickly than smaller particles with smaller settling velocities. In elutriation, the air flows upward so that particles with setting velocities equal to or less than the air velocity will have a net velocity upward and particles which have settling velocities greater than the air velocity will move downward.

There are a number of commercial devices and methods having varying requirements of dust amounts and giving different ranges of size distributions, with a minimum size usually no smaller than two micrometers.[68,69] Disadvantages include the inability of most sedimentation and elutriation devices to give good size resolution, and the length of time (sometimes several hours) required to use some of the methods.

Instruments used for sedimentation include the pan balance, which weighs the amount of sediment falling on it from a suspension, and the pipette, which collects the particles in a small pipette at the base of a large chamber. The Cahn electronic microbalance, (Cahn Instrument Company, 7500 Jefferson St., Paramount, CA 90723), has an attachment that permits it to function

as a settling chamber. Perhaps the most popular elutriator is the Roller particle size analyzer illustrated in Figure 31, available from the American Standard Instrument Co., Inc., Silver Spring, MD). A recent instrument that measures the size distribution of particles in a liquid suspension is the Xray SediGraph, (Micromeritics Instrument Corporation, 800 Goshen Springs Road, Norcross GA 30071), which has a reported range of sensitivity of 0.1 to 100 μm.

Centrifuges--

Aerosol centrifuges provide a laboratory method of size-classifying particles according to their aerodynamic diameters. The advantage over elutriators is that the settling, or precipitation, process is speeded up by the large centrifugal acceleration and that smaller particles may be sized. The sample dust is introduced in the device as an aerosol and enters a chamber which contains a centrifugal force field.

In one type of aerosol centrifuge, the larger particles overcome the viscous forces of the fluid and migrate to the wall of the chamber, while the smaller particles remain suspended. After the two size fractions are separated, one of them is reintroduced into the device and is fractionated further, using a different spin speed to give a slightly different centrifugal force. This is repeated as many times as desired to give an adequate size distribution. One of the more popular lab instruments using this technique is the Bahco microparticle classifier, which is illustrated in Figure 32, and is available commercially from the Harry W. Dietert Company, Detroit, Michigan. The cutoff size can be varied from about two to fifty micrometers to give size distribution characterization of a 7 g or larger (usually 10g) dust sample. A similar instrument is the B.C.U.R.A. (British Coal Utilization Research Association, Leatherhead, Surrey, U.K.) centrifugal elutriator which has a range of four to twenty-six micrometers.[70]

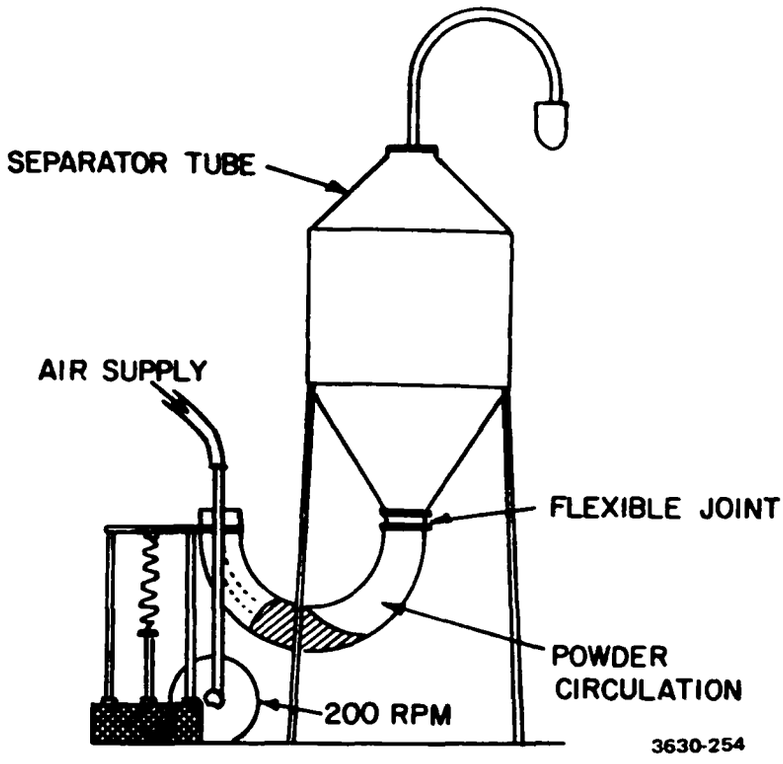
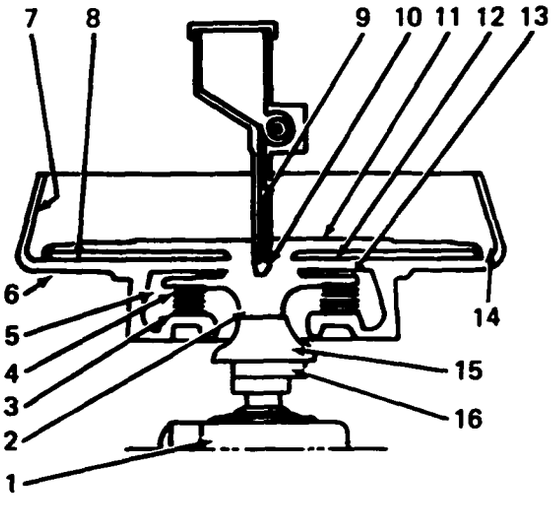


Figure 31. The Roller elutriator. After Allen.[69]



SCHEMATIC DIAGRAM

1. Electric Motor
2. Threaded Spindle
3. Symmetrical Disc
4. Sifting Chamber
5. Container
6. Housing
7. Top Edge
8. Radial Vanes
9. Feed Point
10. Feed Hole
11. Rotor
12. Rotary Duct
13. Feed Slot
14. Fan Wheel Outlet
15. Grading Member
16. Throttle

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Figure 32. The Bahco microparticle classifier.

In the second type of centrifuge, the device is run continuously, and the particle size distribution is determined from the position where the particles are deposited. Examples are a spiral centrifuge developed by Goetz, et al.,[71,72,73] (Figure 33) and by Stöber and Flachsbart [74] (Figure 34) that can classify polydisperse dust samples with particles from a few hundredths of a micron to approximately two microns in diameter. The conifuge, first built by Sawyer and Walton[75] and modified several times since then,[76,77] is useful in the study of aerodynamic shape factor, but can also be used for the determination of size distributions, especially for particles having aerodynamic diameters smaller than twenty-five micrometers (see Figure 35). In continuously operating centrifuges, the particles are generally deposited onto a foil strip, where their position yields a measure of their size, and their number is obtained by microscopy or radiation or by weighing segments of the foil.

Microscopy--

Microscopic analysis has long been regarded as the established, fundamental technique of counting and sizing particles that the human eye cannot comfortably see. Usually, the method involves one person, a microscope, and a slide prepared with a sample of the aerosol to be measured. A random selection of the particles would then be measured and counted, with notable characteristics of color, shape, transparency, or composition duly recorded. The most difficult task, especially since the advent of sophisticated computerized equipment has made counting and sizing easier, is the preparation of a slide which contains a representative sample of the aerosol.

Particle sizes which can be easily studied on optical microscopes range from about .2 to 100 micrometers. Electron microscopes have increased the size range of particles capable of being

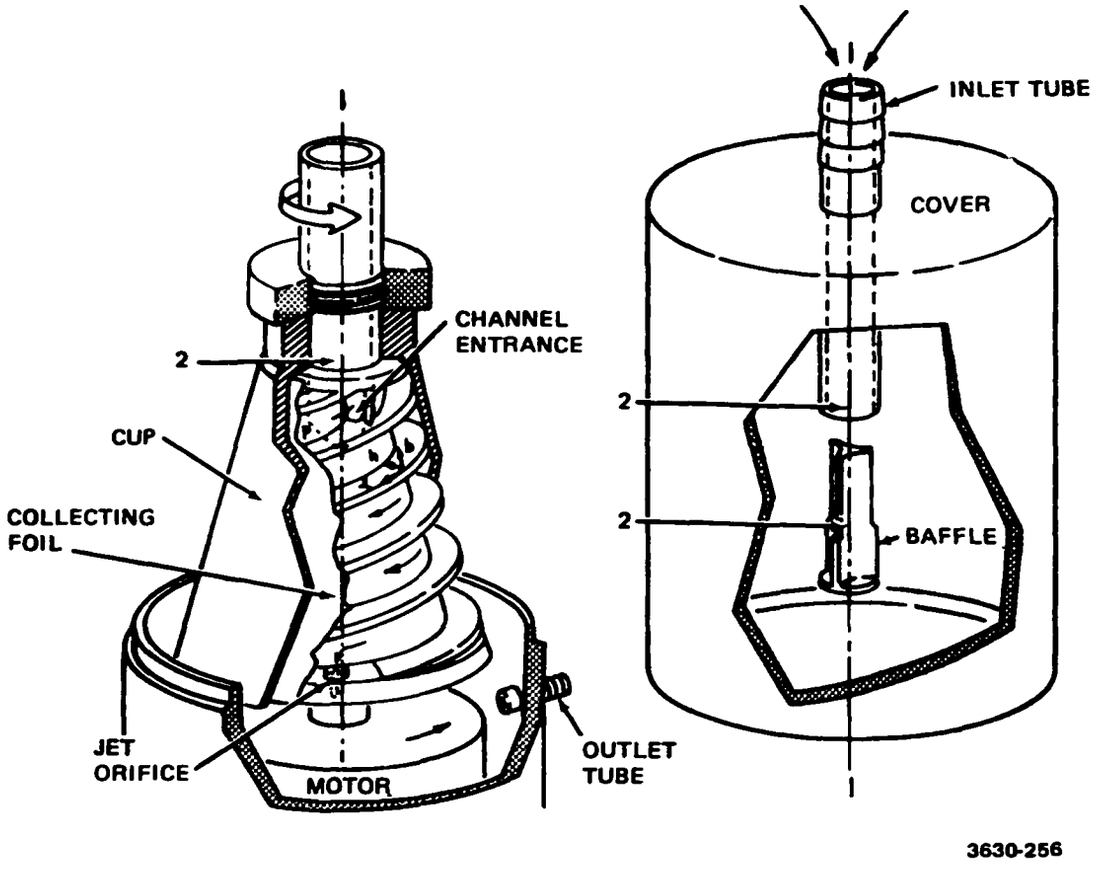

Figure 33. A cut-away sketch of the Goetz Aerosol Spectrometer spiral centrifuge. In assembled form the vertical axes (1) coincide and the horizontal arrows (2) coincide. After Gerber.[73]

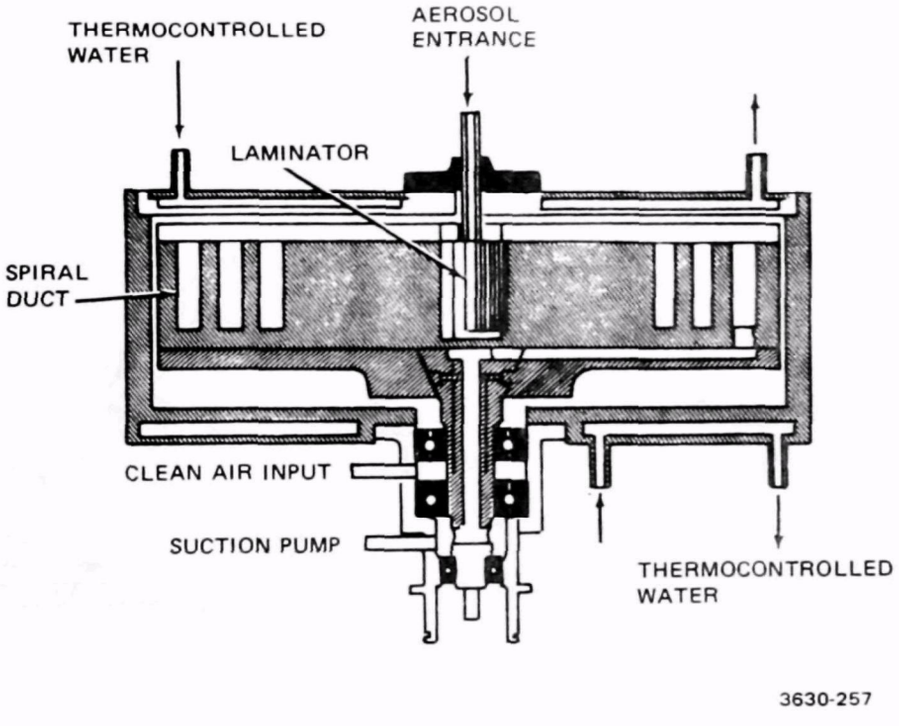

Figure 34. Cross-sectional sketch of the Stober Centrifuge. After Stober and Flachsbart.[74]

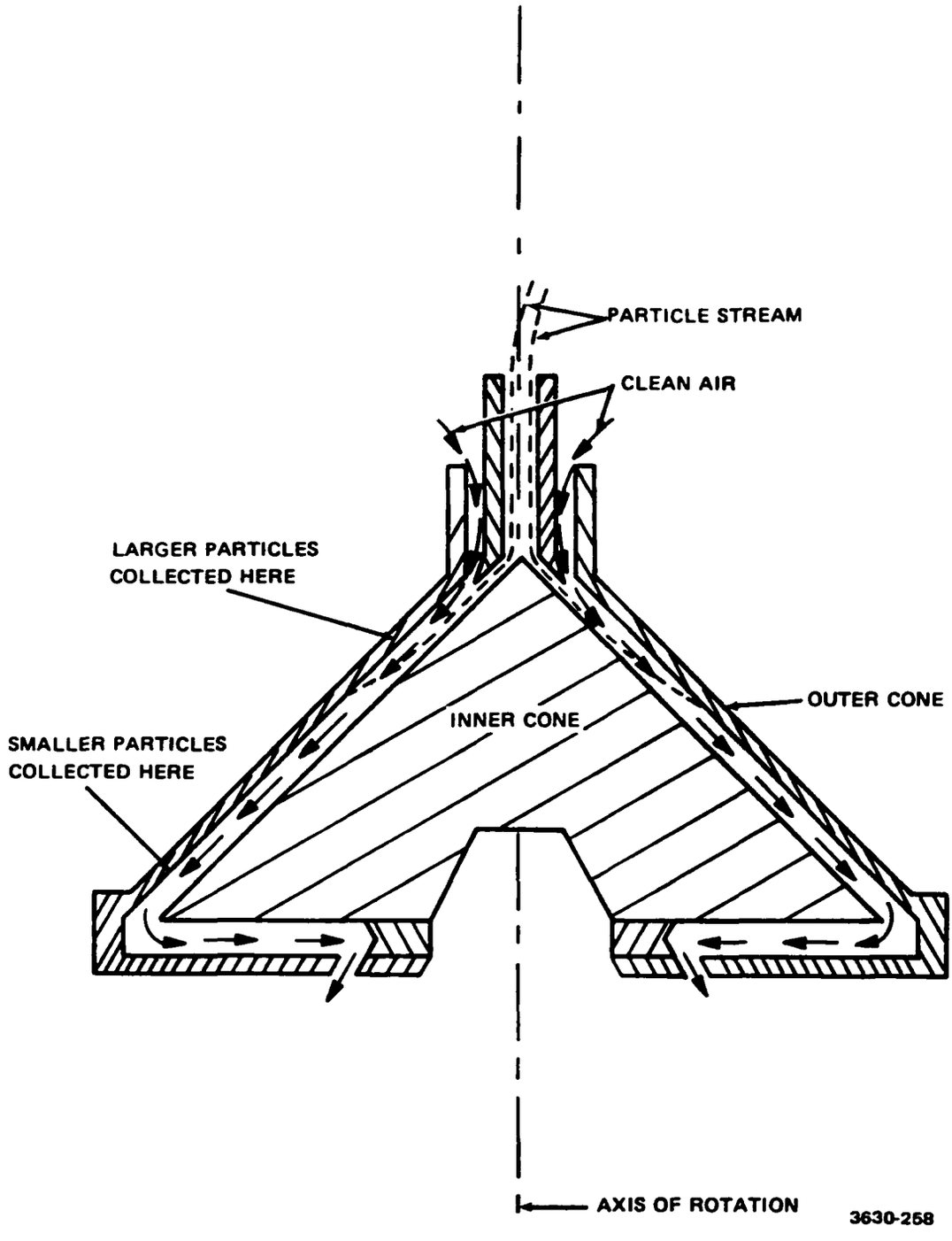

Figure 35. Cross-sectional sketch of a conifuge.

analyzed by microscopy down to 0.001 micrometers. Computerized scanning devices have increased the analyzing ability of present day microscopes and simplified counting and sizing. Several commercial laboratories are equipped to provide physical and structural characterizations of dust samples quickly and fairly inexpensively.

Sieves--

Sieving, one of the oldest ways of sizing particles geometrically, is the process by which a polydisperse powder is passed through a series of screens with progressively smaller openings until it is classified as desired. The lower size limit is set by the size of the openings of the smallest available screen, usually a woven wire cloth. Recently, micro-etched screens have become available. In the future, the lower size limit may be lowered by using membrane filters which can be made with smaller holes than woven fine wire cloth.

Woven wire sieves are available from several manufacturers in four similar standard size series: Tyler, U.S., British, and German. Tyler screens are manufactured by the W.S. Tyler Co., Cleveland, Ohio.

Other methods of size classification using sieving principles are currently being studied and improved. Wet sieving is useful for material originally suspended in a liquid or which forms aggregates when dry-sieved. Air-jet sieving, where the particles are "shaken" by a jet of air directed upward through a portion of the sieve, has been found to be quicker and more reproducible than hand or machine sieving, although smaller amounts of powder (5 to 10 g) are generally used. Felvation[78] (using sieves in conjunction with elutriation) and "sonic sifting"[79] (oscillation of the air column in which the particles are suspended in a set of sieves) are similar techniques that employ this principle.

Because of its relatively large lower particle size limit (50-75 micrometers for woven wire screens), sieving has a limited use for characterizing most industrial sources today. However, for particles within its workable size range, sieving can be a very accurate technique, yielding adequate amounts of particles in each size range for thorough chemical analysis.

Coulter Counter--

Figure 36 illustrates the principle by which Coulter counters (Coulter Electronics, Inc., 590 West 20th Street, Hialeah, FL 33010) operate. Particles suspended in an electrolyte are forced through a small aperture in which an electric current has been established. The particles passing through the aperture displace the electrolyte, and if the conductivity of the particle is different from the electrolyte, an electrical pulse of amplitude proportional to the particle-electrolyte interface volume will be seen. A special pulse height analyzer is provided to convert the electronic data into a size distribution. A bibliography of publications related to the operation of the Coulter counter has been compiled by the manufacturer and is available on request.

NEW TECHNIQUES

Promising instruments and techniques for particle size deterterminations in industrial process streams are summarized in this section. These devices have not had widespread usage under field conditions, and some of them exist only in prototype form. Special skills are needed to operate the instruments and to avoid the problems associated with their use in industrial process streams.

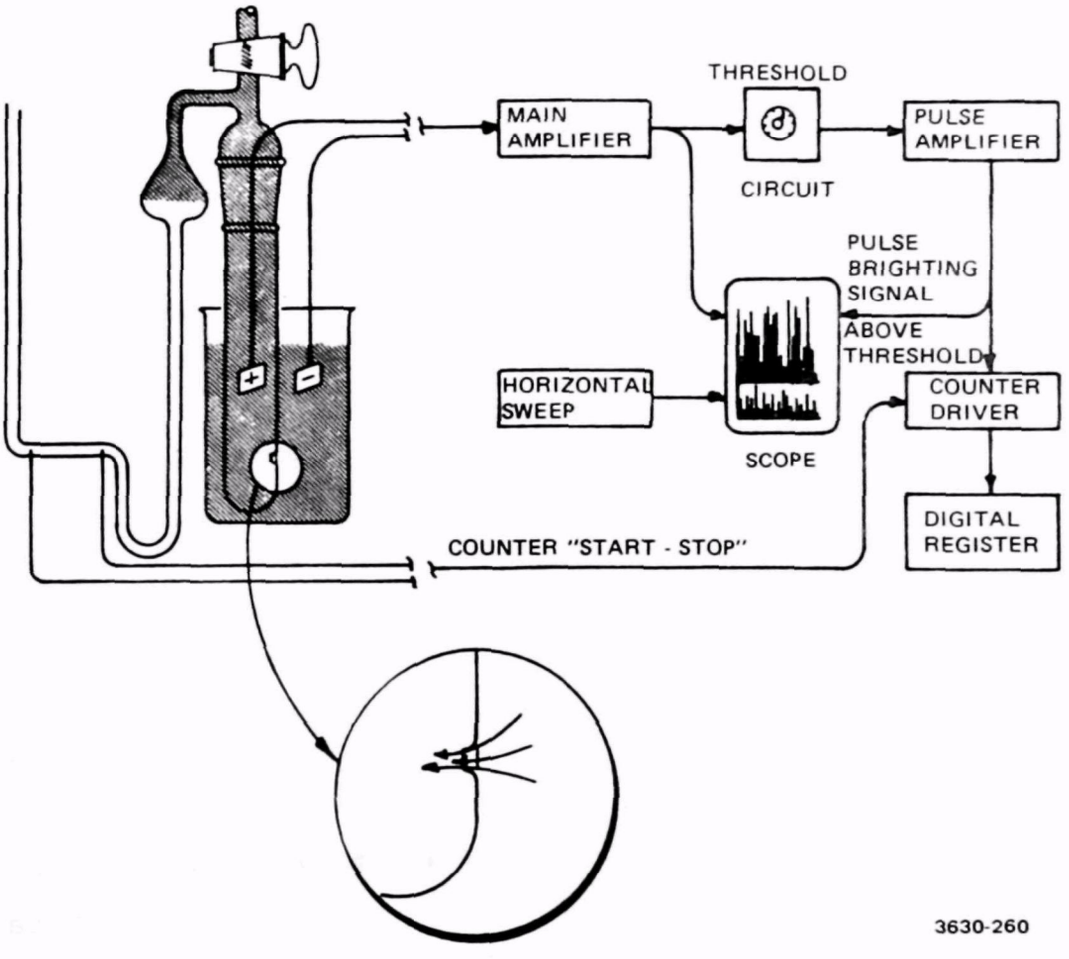

Figure 36. Operating principle of the Coulter counter. Courtesy of Coulter Electronics.

Low Pressure Impactors

It is possible to extend the sizing capability of cascade impactors to submicron particles by operating the device at pressures of 0.01 to 0.1 atmospheres. Pilat[80,81] has developed and tested a low pressure impactor for sampling from process streams. (See Figure 37).

Figure 38 shows the sampling train used by Pilat. Two impactors are operated in series. The first impactor is a conventional design with cut points from about 0.3 to 20 μm diameter, and the second impactor is operated at reduced pressure with cut points from about 0.03 to 0.2 μm diameter. The maximum flow rate is approximately 50 liters/minute. The main problems associated with this technique are the bulky equipment required, the potential for particle bounce, and the very low mass collected on each stage.

Impactors with Beta Radiation Attenuation Sensors

Beta attenuation has some appeal as a detection mechanism for cascade impactors in air pollution work because the impactor separates the particles according to their aerodynamic behavior, and the beta attenuation yields a direct, nearly real-time measure of the amount of mass collected. However, the unavailability of high temperature beta detectors has hindered attempts to develop in situ instruments. Other problems include: selecting suitable tapes and greases for compatibility with the beta monitor and for good particle retention, designing the impactor to give a uniform deposit, and the mechanical problems associated with designing such a complex system to be operated in a harsh, dirty environment. It is unlikely that multiple stage impactors with beta attenuation as detection mechanism can be made practical for in stack use in the foreseeable future.

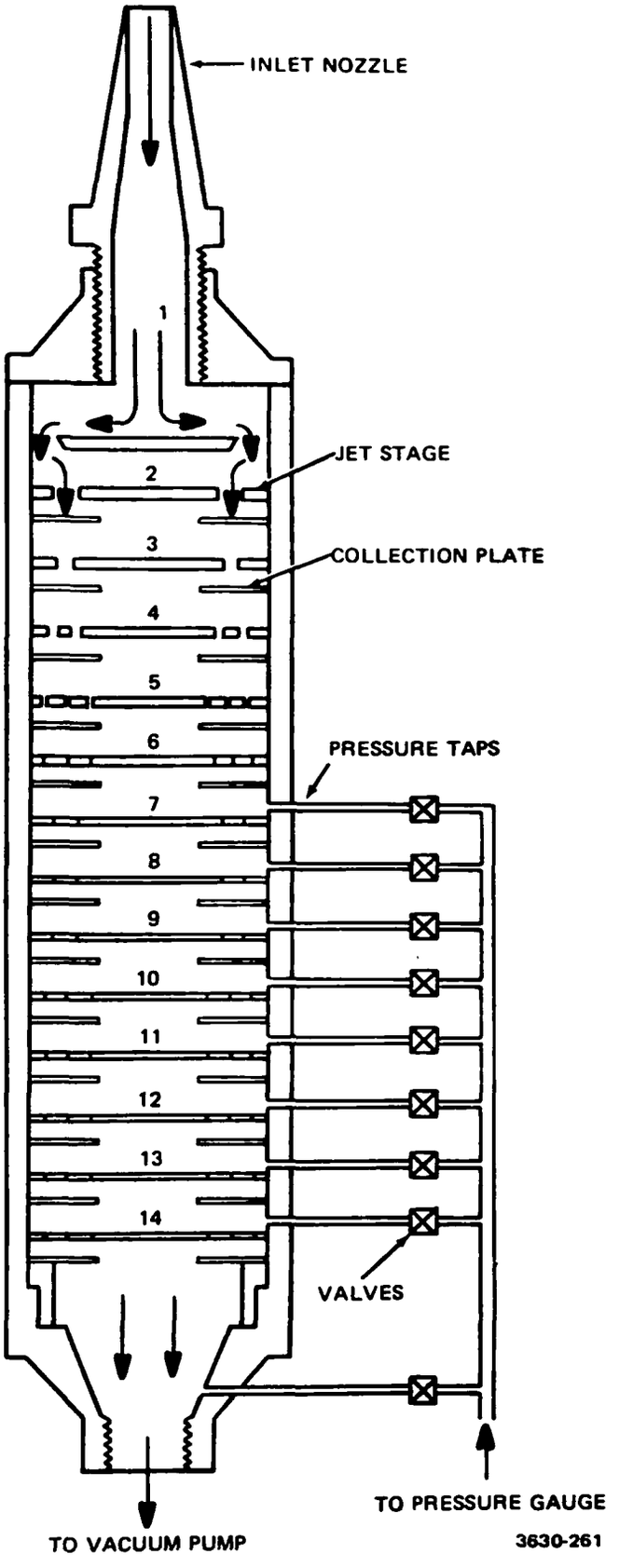

Figure 37. Cross section of prototype Mark IV University of Washington Source Test Cascade Impactor.

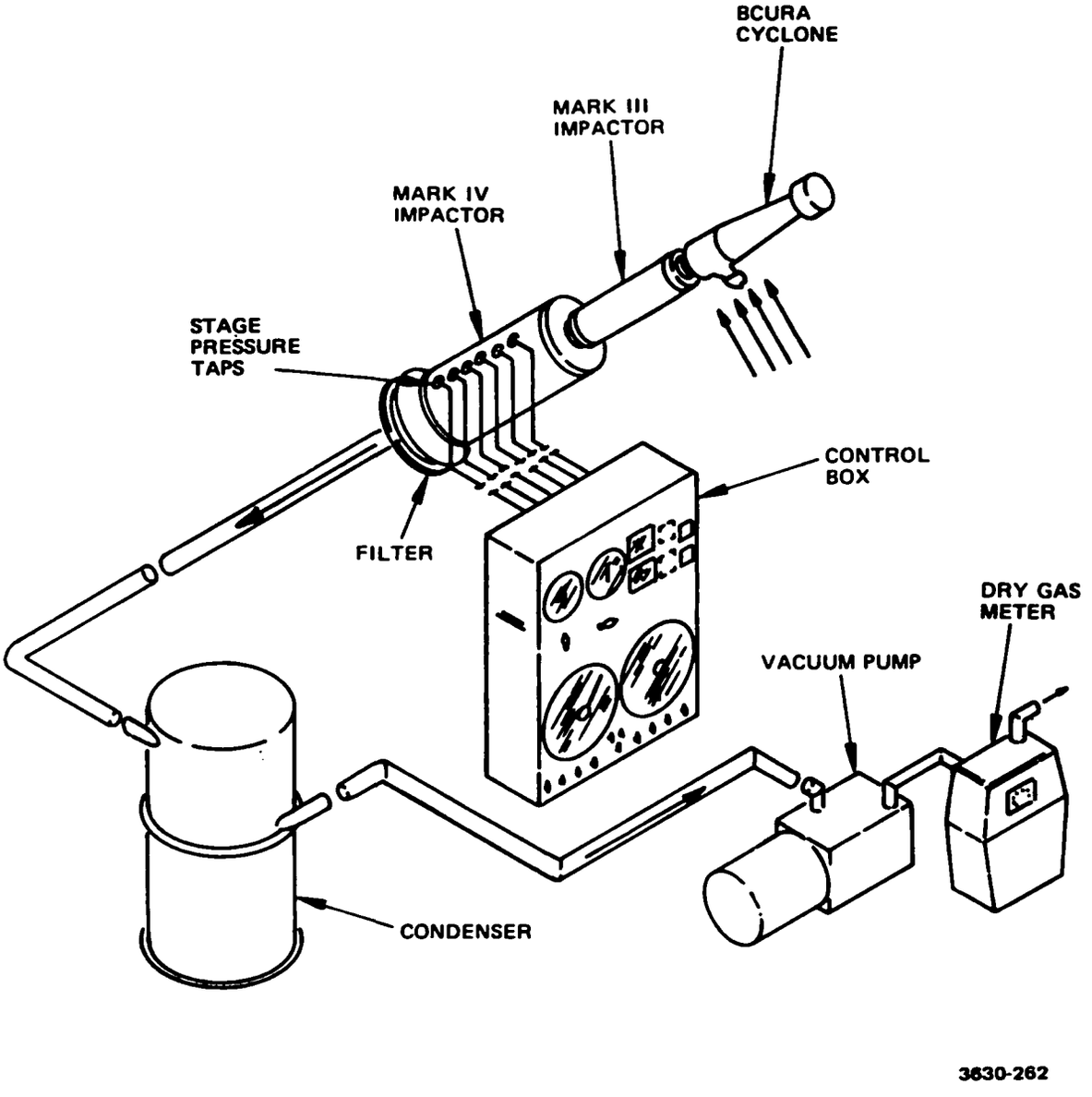

Figure 38. Sampling train utilizing a low pressure impactor. After Pilat.81

Cascade Impactors with Piezoelectric Crystal Sensors

Carpenter and Brenchly[82] and Chuan[83] have developed and tested multiple-stage cascade impactors with piezoelectric crystals on each stage to monitor the rate and amount of mass collected. Chuan's impactor is now sold commercially by Berkeley Controls, Inc. (2700 Du Pont Drive, Irvine, CA 92714). Chuan's impactor has ten stages, with the cut points reported to be from 0.05 to about 25 μm. Because of the extreme sensitivity of the instrument (and upper limit on mass accumulation), it is more suitable for ambient than stack work, where sample extraction and dilution would be required. The best application of piezoelectric impactors would seem to be monitoring real time fluctuations in fairly dilute aerosols. For more information, see Piezoelectric Mass Monitors under Mass Concentration.

Virtual Impactors

Figure 39 illustrates the operating principle of virtual impactors, sometimes called centripeters, dichotomous samplers, or stagnation impactors. The aerosol jet is directed toward a stagnant zone, or an opposing jet of clean gas, and a "virtual" surface is formed at the boundary between the aerosol jet and air space or opposing jet. The jet streamlines are diverted as in a normal impactor. Particles of larger Stokes number impinge on (and pass through) the virtual surface, while those having smaller Stokes numbers follow the streamlines.

Several multiple-stage virtual impactors have been developed, all for the purpose of obtaining large quantities of sized particles, in uniform deposits, for subsequent analysis. Hounam and Sherwood,[84] Conner,[85] Peterson,[86] and Loo, et al.[87] have developed virtual impactors with BGI, Inc. (58 Guinan St., Waltham, MA 02154) handling the Hounam and Sherwood version and Sierra

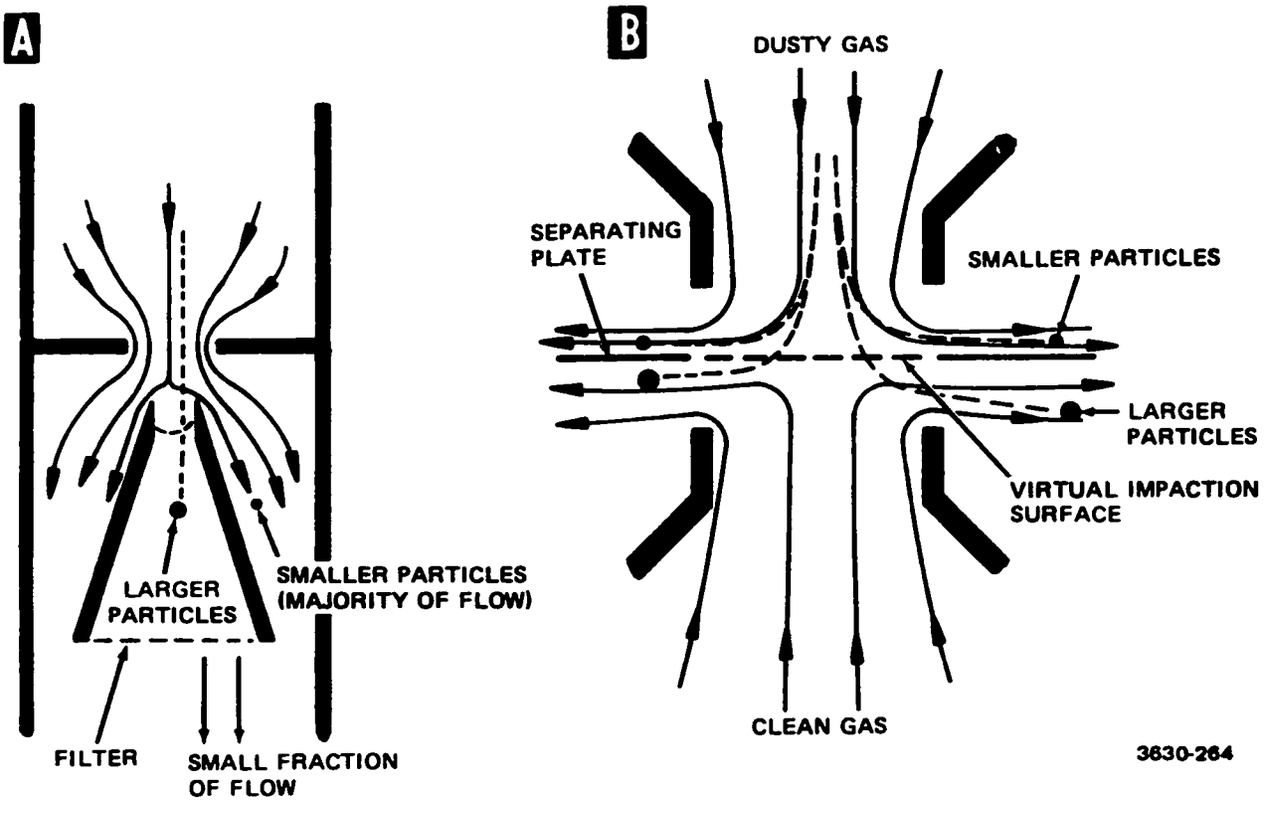

Figure 39. Virtual impactors (centripeters, dichotomus samplers, stagnation impactors) a. impingement into a stagnant air space; b. opposed axisymmetric jets.

Instruments Co. (P.O. Box 909, Village Sq., Carmel Valley, CA 93924) handling Peterson's version. Since the performance of opposed-jet impactors appears to be quite sensitive to the geometry and alignment of various components, a rugged field model is not yet available.

Virtual impactors have played a minor role in pollution studies to date, with very little, if any, application to process streams. The major advantage of these devices appears to be the capability of using them to obtain large samples, apparently an absence of particle reentrainment and uniformly deposited films of dust for analysis by X-ray fluorescence, or any other technique that requires similar sample preparation.

Optical Measurement Techniques

When light is incident upon a particle, some of the radiation will be absorbed, some scattered, and some polarization will occur. The exact nature and magnitude of the interaction depends on the ratio of the particle diameter to the wavelength of the radiation, and the shape and composition of the particle. Thus, measurements can be envisioned that would yield information of particle size, shape, concentration, and composition. It appears, from the information now available, that optical methods offer the greatest hope for a major advance in the technology of particulate sampling. Any successful instrument, however, must be able to function in a harsh environment where extremes in temperature, particle concentration, corrosion, etc. are found. Also, the parameter that is measured should ideally be related to the aerodynamic diameter of the particles.

Although there are no proven commercial instruments available for measuring particle-size distributions in process streams, a variety of methods have been proposed, and several prototype instruments developed.

Hodkinson[88] suggested a method of minimizing the dependence on particle refractive index in sizing measurements from a study of the Fraunhofer diffraction formulation at small angles of forward scattering. The basis of this method involves measurement of the intensity of light scattered by a single particle at two small angles, and calculation of the ratio of the two intensities.

Shofner, et al.,[89] Gravatt,[90] and Chan[91] have developed prototype systems for particle sizing that are based on the intensity ratio concept of Hodkinson. Shofner's system, the "PILLS-IV", is designed for in situ operation. The useful size range for particle sizing is from 0.2 to 3.0 μm diameter. Shofner states that the view volume of his system is approximately 2×10^{-7} cm^3. The upper concentration limit for single particle counters is determined by the requirement that the probability of more than one particle appearing in the view volume at a given time be much less than unity. For Shofner's system this would set the concentration limit at approximately 10^6 particles/cm^3, a value much higher than for conventional single particle counters.

A real time in situ particle sizing probe utilizing small angle light scattering is being developed for stack use under the sponsorship of the U.S. EPA.[92] It is an adaptation of an optical particle sizing device developed for atmospheric measurements. The field prototype of the instrument is now being tested. The instrument covers a 0.3 to 10.0 μm size range with 60 channels resolution. The major uncertainty in sizing spherical particles with the instrument, performed by relating size to flux scattered at small forward angles by single particles, is the particle refractive index giving at most an error of ±20% and normally within ±10% of actual size. The maximum concentration for accurate measurements limited by coincidence counting in the

present model is $5x10^4$ cm^{-3}. Normally, the main effect of higher concentrations is to decrease the effective size range. An optical velocimeter is also designed into the instrument. The present design permits temperatures up to 250°C and velocities up to 30 m/sec. The results of an initial in-stack test at a coal-fired power plant with an ESP and a scrubber were reasonable. Calculated opacity from the measured particle size distribution was about 15% while measured opacity was 17%. The calculated mass loading was 0.01 to 0.02 gm/m^3 with a volume average diameter of about 1.3 μm. It appears that the instrument was capable of resolving several size modes in this test.

Systems employing optical Fourier transforms to obtain particle-size distributions in the 5-100 μm diameter range have been described by Cornillaut[93] and McSweeny.[94] With the proper selection of measurement points in the diffraction pattern the size interval covered by the technique can be extended outside the previously mentioned 5-100 μm range.

Another in situ portable light scattering instrument being developed under EPA sponsorship to determine size distribution utilizes diffraction and polarization from scattering.[95] This device measures flux scattered from many particles simultaneously at three small angles relative to the forward direction, 4°, 8°, and 11°, and at a range of large angles 80°-100°. Each measurement is performed at two wavelengths, 0.45 and 0.9 μm, and the large angle scattering is measured at two orthogonal polarizations. The instrument relates the small angle signals dominated by Fraunhoffer diffraction to the volume of particles in three size ranges centered at 1.0, 3.5, and 7.0 microns. For the lower end of the size distribution, the differences in the two 90° signals at two orthogonal polarizations obtained with the 0.9 μm (0.45 μm) wavelength is related to the volume of particles in a size range centered about 0.4 μm (0.2 μm). The size, range, mass loading, and temperature ranges are 0.1 to 10 μm, 4 to 400 ppb by volume, and 0° to 260°C. The prototype has been delivered to EPA to be tested in a wind tunnel facility.

Imaging systems, either of a direct type or of a type using reconstructed images from holograms, have not been widely used for size distribution analysis in flue gases but have been used routinely for work with liquid aerosols.

Flash television particle counters providing real time size distributions have been described by Hotham[96] using pulsed ultraviolet laser illumination and by Simmons and Dominic[97] using xenon flash tubes for illuminators. The reported range for size distribution determinations for the latter device is 0.3 to 10,000 μm. Because of cost and practical difficulties involved in the use of such a system in a flue gas environment, applications of these systems will probably be limited to special research applications.

Holography as a technique for investigating aerosols has several advantages over most of the methods previously described. The aerosol is not disturbed by the measurement process, a large depth of field is possible and, as in the flash television method, the particles can be effectively "stopped" for examinations at speeds up to a few hundred meters per second. Typical system resolution limits, however, result in a lower limit in sensitivity for particle sizing of about 5 μm. By double-pulsing the laser illuminator one can obtain holograms which permit the determination of particle velocities in three dimensions. Image Analyzing Computers, Inc., of Monsey, NY, offers an automatic analyzer for reading out and analyzing aerosol data from holograms, making it possible to eliminate manual analysis.

Laser Doppler Velocimeters (LDV) are routinely used for measuring the velocity of gases, and these instruments can also be used to obtain information on particle size. Farmer,[98] Robinson and Chu,[99] Adrian and Orloff,[100] and Roberds[101] have done experimental and theoretical studies of LDV systems designed to enhance

the sensitivity to particle size. A commercial LDV particle spectrometer based on Farmer's work is available from Spectron Development Laboratories, Inc. (Tullahoma, TN 37388). Advantages of LDV systems are the potential for in situ sampling with little or no perturbation of the sample. Disadvantages are the sensitivity to particle refraction index and complexity of the system.

Hot Wire Anemometry

An electronic instrument has been developed by Medecki, et al.[102] of KLD Associates, Inc. (Huntington, NY, USA) for sizing liquid droplets, especially in scrubbers. The instrument operates by inertial deposition of 1 μm to 600 μm spray droplets on a 5 μm diameter by 1 mm long platinum sensing element of the type used in hot-wire anemometry. Droplets smaller than 1 μm can be measured with a change in sensor geometry. The sensing element is electrically heated to a predetermined temperature. Impinging particles cool the sensing element, resulting in changes in resistance which are related to the sizes of the impinging droplets. The commercially available version of the device provides concentration outputs in six selectable size channels. Size calibrations for the channels are for water droplets; however, the application of the method is not, in principle, limited to water. Because the device is essentially a modification of a hot-wire anemometer, it could also theoretically be used to measure flow velocity and temperature permitting impingement rates to be converted to aerosol concentrations. Although commercial prototypes are available now, this instrument is still under development and detailed performance analyses are not available.

Large Volume Samplers

McFarland and Bertch[103] have developed a system for collecting bulk samples of classified dust for subsequent use in health related research. The system contains, in series, two cyclones,

a virtual impactor, and a bag filter. The D_{50}'s of the cyclones are 10 and 7 μm, and that of the virtual impactor is 5 μm at a sample flow rate of 850 l/min. The particulate collection components are housed in an insulated enclosure that is 2.7 x 1 x 2 m. In sampling for 12 days at the outlet of an electrostatic precipitator, McFarland collected 8.1 kg of dust: 5.4 kg in the large cyclone, 1.3 kg in the small cyclone, 0.6 kg in the virtual impactor, and 0.8 kg in the filter. A new system, designed to sample at a flow rate of 33 m^3/min is now under development.[104]

SECTION V
CONTROL DEVICE EVALUATION

Several reasons exist for performing control device evaluations. These reasons may range from a verification of compliance with emissions requirements to programs related strictly to research.

The majority of stationary air pollution sources need some type of control device to satisfy the national, state, or local air pollution regulations that limit the allowable emissions. In order to determine whether the plant is in compliance with these regulations, tests are performed to measure the amount of air pollutant emissions from the control device in question. This is one type of control device evaluation and it is usually the simplest and least expensive.

Another reason for performing tests on a control device is to optimize the performance of the installation. These tests might be requested by the owners of the plant where the control device is installed, or by the control device manufacturer. Usually tests of both the inlet and outlet particulate mass concentration are made resulting in a measure of the particulate collection efficiency. In some instances the fractional efficiency (efficiency as a function of particle size) is desired and measurements of the particle size distributions of the inlet and outlet dusts are necessary.

If a particular control device is performing poorly due to poor maintenance, or poor design, etc., then tests might be required in order to obtain data to be used in designing additional or replacement control device units.

To obtain data for purely research purposes is a fourth reason for performing a control device evaluation. In each test the data may be used to confirm existing theories of control device operation or to develop new theories for modelling and predicting control device performance. Research tests may involve total systems studies on the source/control device combination. These tests are usually the most complicated and expensive because of the amount of data that is desired.

Table VI indicates some of the considerations and problems that must be dealt with in developing a test plan for control device evaluations. Although this table is designed to serve as a planning outline, the relative importance of the facets of the plan, or considerations that are not listed, can only be established from a good understanding of the plant-control device system and the objectives of the test.

A more detailed treatment of control device evaluations can be found in Procedures Manual for Electrostatic Precipitator Evaluation, EPA-600/7-77-059, and Procedures Manual for Fabric Filter Evaluation, EPA-600/7-78-113, available from the National Technical Information Service, Springfield, Virginia.

TABLE VI

PARTICULATE CONTROL DEVICE TASKS

Objective of Tests	Assure Compliance with EPA Regulation	Optimize Performance of Control Device	Obtain Design Data for Control Device	Obtain Data for Modeling Studies	Systems Studies Process and Control Device
Tests Required					
Mass Concentration	O	O	I	I,O	I,O
Opacity	O	O			O
Gas Composition	O	O	I	I,O	I,O
Gas Temperature			I	I,O	I,O
Gas Volume	O		I	I,O	I,O
Pressure			I	I,O	I,O
Velocity Distribution		C	C	X	X
Particle Size Distribution			I	I,O	I,O
Dust Composition			Qualitative	Qualitative	I,O*
Dust Resistivity				ESP only	X
Control Device Data					
Operating				X	X
Design				X	X
Maintenance					X
Plant Process Data				D	D
Economic Data				Optional	X
Technical Considerations (Decisions/Problems)					
Adequate Space, Electric Power	O	O	I	I,O	I,O
Laboratory Space	X	X	X	X	X
Number of Tests Required	3	D	D	D	D
Isokinetic Sampling	X	X	X	X	X
Condensible Vapors/ Volatile Particles		C	C	C	C
Mass Concentration/ Sampling Time	C	C	C	C	C
Traverse Strategy	O	O	O	O	O
Aerosol Gas Velocity	C	C	C	C	C
Process/Emission Variations	C	C	C	C	C
Select Particle Sizing Methods			I,O	I,O	I,O
Select Mass Train Type	O	O	I,O	I,O	I,O
Select Gas Analysis Methods		D	D	D	D
Real-Time Monitors Needed		D	D	D	D
Filter Mass Stability		C	C	C	C
Sample Preservation		D	D	D	D

Key:
- O Outlet
- I Inlet
- X Required
- D Decision based on specific site or test objectives
- C Must be considered
- * vs. Particle Diameter

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An extensive search was made of the references in the Environmental Engineering Library of Southern Research Institute, which contained a major portion of the references listed in the bibliography. Publications over three years old were generally not included unless they contained information that was not found or not superceded in recent papers. The list of references cited in Sections II - IV of the manual are not necessarily duplicated in the bibliography, however they should be consulted first for information on particulate measurement.

The formats of the references generally fall into four groups:

1. Reports on government contracts: authors, title, performing organization or company, sponsoring government agency, address of government agency, year of publication,

number of pages, government report number (when applicable or available), and National Technical Information Service number (when applicable or available).

2. Books: authors, title, publisher, publisher's address, year of publication, and number of pages.

3. Journal articles: author(s), title, name of journal, volume number, issue number (if applicable), page numbers, and year of publication.

4. Papers and proceedings of technical meetings: author(s), title, name, location, and year of meeting, page numbers or paper number (when applicable).

Contents of the bibliography are arranged alphabetically by author under the following headings:

1. General References
2. Sample Extraction
3. Filter Media
4. Mass Concentration
5. Particle Size Distribution
6. Opacity - Transmissometers - Nephelometers
7. Analytical Technique
8. Control Device Evaluation - Field Tests.

A more extensive bibliography can be found in the companion document Technical Manual: A Survey of Equipment and Methods for Particulate Sampling In Industrial Process Streams, EPA-600/7-78-043, March, 1978, by Wallace B. Smith, Paul R. Cavanaugh, and Rufus R. Wilson.

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16. ABSTRACT The report lists and briefly describes many instruments and techniques used to measure the concentration or size distribution of particles suspended in process streams. Standard (well established) methods are described, as well as some experimental methods and prototype instruments. Instruments and procedures for measuring mass concentration, opacity, and particle size distribution are described. Procedures for planning and implementing tests for control device evaluation are also included.

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