United States
Environmental Protection
Agency

Health Effects Research
Laboratory
Research Triangle Park NC 27711

EPA-600/1-80-014
February 1980

Research and Development

EPA

Carbonaceous Aerosol Generator for Inhalation Studies

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CARBONACEOUS AEROSOL GENERATOR FOR INHALATION STUDIES

by

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Interagency Agreement No.
EPA - 78-D-X0170

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FOREWARD

The many benefits of our modern, developing, industrial society are accompanied by certain hazards. Careful assessment of the relative risk of existing and new man-made environmental hazards is necessary for the establishment of sound regulatory policy. These regulations serve to enhance the quality of our environment in order to promote the public health and welfare and the productive capacity of our nation's population.

The Health Effects Research Laboratory, Research Triangle Park, conducts a coordinated environmental health research program in toxicology, epidemiology, and clinical studies using human volunteer subjects. These studies address problems in air pollution, non-ionizing radiation, environmental carcinogenesis and the toxicology of pesticides as well as other chemical pollutants. The Laboratory participates in the development and revision of air quality criteria documents on pollutants for which national ambient air quality standards exist or are proposed, provides the data for registration of new pesticides or proposed suspension of those already in use, conducts research on hazardous and toxic materials, and is primarily responsible for providing the health basis for non-ionizing radiation standards. Direct support to the regulatory function of the Agency is provided in the form of expert testimony and preparation of affidavits as well as expert advice to the Administrator to assure the adequacy of health care and surveillance of persons having suffered imminent and substantial endangerment of their health.

A carbonaceous aerosol generator was designed and built for inhalation studies with animals. The aerosol generated will be carbonaceous with size characteristics comparable to the published data on diesel exhaust particles. Such a generator can be used to conduct a variety of studies in animals under carefully controlled conditions.

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Abstract

A carbonaceous aerosol generator designed for inhalation experiments with animals is described. The aerosol produced from a modified diffusion flame has a concentration of 3-10 mg/m^3 at a flow rate of 30 L/min. The addition of a small amount of O_2 to the acetylene fuel greatly increased the efficiency of fuel to particulate conversion, the maximum value was 2.5%. The aerosol size characteristics were: $\overline{D}_{gn}$ = 0.14 μm, based on the electrical aerosol analyzer; $\overline{D} \simeq$ 0.08 μm, based on a low pressure inertial impactor; median elementary particle $\simeq$ 0.023 μm and median agglomerate particle $\simeq$ 0.54 μm, based on transmission electron microscopy. The size characteristics of the generated aerosol are compared with diesel exhaust based on available published data.

Key words: Aerosol generator; agglomerate; diesel exhaust; diffusion flame; inhalation; particulates; particle size; soot

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SECTION 1

INTRODUCTION

The Center for Fire Research, National Bureau of Standards, Department of Commerce entered into an agreement with the Health Effects Research Laboratory, Environmental Protection Agency (EPA) to develop a combustion type carbonaceous aerosol generator and to characterize the resulting aerosol. The generator is to be used by EPA for animal lung deposition studies using C^{14} tagged radioactive fuel.

This report describes the carbonaceous aerosol generator and includes information on test repeatability, the size distribution of the generated aerosol, number and mass concentration of particulates, carbon-hydrogen ratio, and the concentration of CO and CO_2 gases in the aerosol stream. Detailed assembly and operation instructions for the generator are also included in the appendix.

During the development of the generator, EPA added the requirement that the generator have a carbon conversion efficiency of fuel to particulate in the order of 5% or higher and a maximum particulate generation rate of about 0.3 mg/min at a flow of 30 L/min with a variable concentration between 5 and 10 mg/m^3. This need for a high conversion ratio and low output generation became obvious because of the high cost of C^{14} labeled fuels, which are necessary for making lung deposition studies in animals.

A major thrust of the generator development was concerned with meeting this added requirement. Our early work indicated that the 5% conversion efficiency could be obtained using a diffusion flame with propane fuel; however, the particulate generation rate was a factor 20 higher than the desired level. Much of the subsequent work was concerned with finding the best combination of nozzle design, combustion conditions, and additives to yield high conversion of fuel to particulate at a low fuel burning rate. In other words, the goal is to promote sooting in a very small flame. It was found that an acetylene diffusion flame with a small amount of premixed oxygen or N_2O yielded the best result though the efficiency is still somewhat less than the desired value. All subsequent discussion will be confined to acetylene diffusion flame with premixed oxygen.

The second part of this study was concerned with the physical and chemical characterization of the carbonaceous aerosol. Since the carbonaceous aerosol is to be used to simulate the particles of diesel smoke, it is important to know the characteristics of the carbonaceous aerosol so that comparison between the two may be made. The chemical analysis consisted of an analysis of carbon-hydrogen ratio of the particulate and analyses for CO and CO_2 in the gas phase of the aerosol.

All of the techniques used by Dolan et al. [1][1] and Vuk et al. [2] in their physical measurements of the size distribution of diesel particulate were applied in the measurements of the carbonaceous aerosol from the generator, including electrical aerosol analyzer (EAA), scanning and transmission electron microscopy, and cascade impactors. In addition, the aerodynamic size distribution was determined by a low pressure cascade impactor developed by Hering et al. [3] with a 50% efficiency cut-off on the last stage of 0.05 μm, which is almost a factor of 10 smaller than ambient pressure cascade impactors currently available.

[1] Numbers in brackets refer to literature references at the end of this paper.

SECTION 2

CONCLUSIONS AND RECOMMENDATIONS

A stable, repeatable carbonaceous aerosol generator has been developed for animal inhalation studies with a carbon conversion efficiency of fuel to particulate of up to 2.5% at a mass concentration of 10 mg/m^3 and a flow rate of 30 L/min.

The particle size as measured by the EAA, cascade impactors, and electron microscopy and the carbon content in the particulate agree reasonably well with the results based on the high-temperature diesel exhaust.

The median aerodynamic particle size based on the low pressure impactor measurement is about 0.08 μm for the generated aerosol. This is perhaps the first aerodynamic classification of a carbon agglomerate type aerosol in the size range below 0.2 μm.

If the conversion efficiency of the present generator is shown to be too low for long-term economic use with a radioactive tagged fuel, there are two other alternative generator designs that might yield higher efficiency. Details for their designs are presented in the discussion section of the report. A second area where additional research is needed concerns the characterization of the size distribution and shape of the carbonaceous aerosol. The advantages of using a low pressure impactor and a diffusion battery for such a study are also presented in the discussion section of the report.

SECTION 3

DESIGN OF CARBONACEOUS AEROSOL GENERATOR

DESIGN AND CONTROLS

The carbonaceous aerosol generator was designed to generate a low concentration of carbonaceous aerosol (5 to 10 mg/m^3) so as to be suitable for animal inhalation experiments without further dilution though at the same time yielding a high conversion ratio of fuel to aerosol. The generator design allows considerable flexibility in the choice of combustion conditions including type of fuel, diameter of burner nozzle, use of fuel additives and diffusion air, and the temperature of the combustion chamber.

The generator consists of three parts: combustion chamber, control panel, and monitoring photometer.

COMBUSTION CHAMBER

The inside and outside views of the combustion chamber are shown in figures 1 and 2. The fuel enters the burner through the bottom of the chamber at a flow rate of about 15 cm^3/min. The 1.7 mm-ID nozzle of the brass burner is located 20 mm above the base of the chamber. The diffusion air mixture enters through a sintered porous bronze cylinder, 6 mm thick, at a flow rate of about 1 L/min. In the final design a small amount of O_2, about 5 cm^3/min, is premixed with the acetylene fuel. Diffusion air is diluted with nitrogen to a final mixture of about 16% oxygen in order to increase the efficiency of soot production. The combustion is initiated by a spark generated from a high voltage wire located next to the burner tip.

The pressure in the combustion chamber was maintained at about 29 Pa (0.3 cm H_2O) above ambient with an exit orifice to prevent the high volume of dilution air from affecting the generation rate of aerosol. Eight 1.4 mm-ID holes were drilled on the circumference of the tee fitting located between the combustion chamber exit and the dilution air line in order to minimize the wall loss of the aerosol. Apparently the high velocity aerosol exiting from the nozzle entrained air through these holes and thus minimized the wall loss of the particles at the critical area.

The entire stainless steel combustion chamber was heated and maintained at a temperature of about 40° C by eight small heaters embedded in thick chamber walls. It was found that the temperature control of the chamber was necessary to minimize long term drift in aerosol generation rate. Heating of the chamber wall by the flame is a major cause for the drift.

CONTROL PANEL

The key features of the control panel are shown in figure 3. Flowmeters (rotameter) are used for monitoring the flow of fuel, fuel additive, air and N_2. Because of the small flow rates for fuel and fuel additive, a bubble flowmeter is required for accurate calibration and is included with the control cabinet. Calibration curves in figure 4 for the fuel and oxygen flowmeters are based on calibration using a soap bubble flowmeter. Calibration curves in figures 5 and 6 for nitrogen and air flowmeter are based on data supplied by the manufacturer.

One of the three magnehelic gauges on the front of the control panel is used for monitoring the flow rate of dilution air, which may be varied between 8 and 32 L/min by the valve located at the lower right hand corner of the panel. A dilution air flow of 27 L/min, which corresponds to a reading of 0.68 cm H_2O (66 Pa) on the magnehelic gauge, was used for most experiments. A calibration curve for dilution air flow rate versus meter reading for the orifice meter is shown in figure 7.

The second magnehelic gauge is used to monitor the combustion chamber pressure. The middle gauge is coupled to an audible alarm, which can be set to alarm if the chamber pressure is either higher or lower than the preset values.

Other features of the control panel include an ignition button for the fuel, a temperature controller for the combustion chamber, and digital meters to monitor exhaust gas temperature and chamber temperature. The digital meter for monitoring the exhaust temperature is also interfaced to the audible alarm, which will trigger if the temperature drops below 50° C indicative of a flame-out condition. A six foot umbilical cord is provided to allow separation of the burner from the control panel.

MONITORING PHOTOMETER

The monitoring photometer, illustrated in figure 8, is a commercially-available, light-scattering-type smoke detector modified to provide an analog output. The chamber of the detector has also been modified to allow steady-state flow through. The photometer is used for monitoring the mass generation rate and stability over the range from 3 to 12 mg/m^3. A connector on the front of the control panel interfaces the photometer to a recorder.

SECTION 4

EXPERIMENTAL

PERFORMANCE OF GENERATOR

Stability

The output stability of the generator is shown in figure 9 for an aerosol concentration of about 6.5 mg/m^3. The background reading with no smoke present is 0.2 volts as shown in figure 9. The photometer output increases rapidly to about 1.13 volts when the fuel is ignited. It is noted that the output is relatively stable over a 35 minute period though there is a slight downward concentration drift of about 7% after 30 minutes. In earlier work, drift was found to result from an incandescent carbon deposit on the high voltage ignition wire or on the nozzle. By trimming the ignition wire and substituting a brass burner for the stainless steel burner, the deposit no longer formed and the drift was greatly reduced.

Repeatibility

Figure 10 gives an indication of the repeatability of the generator. The generator is on for 15 minutes, off for 10 minutes, and on again for about 10 minutes. It is critical that the fuel and additive needle valves remain in the same position when the burner is turned on and off, because the sooting rate is very sensitive to any change in the flow rate or valve position. Only the on-off toggle valves should be used during the on and off cycling of the generator. To assure good repeatability, the fuel and oxygen lines should be purged for at least 15 minutes if valves upstream of the regulator are shut off.

Figure 11 compares the photometer output for two test runs to show the degree of repeatability and long term (> 30 min) stability of the generator at a high concentration level. The record for run 2A is placed on top of run 2B. The mass concentrations of the aerosol, determined by the filter collection method for a 10 minute sampling period for each run, were 9.1 and 9.3 mg/m^3 for 2A and 2B respectively. Geometric mean sizes of aerosols from the above runs which have been diluted in the 1.8 m^3 chamber are given in table 1.

The results from figure 11 and table 1 show that the generator can provide a reasonably consistent aerosol both in geometric mean size as well as in concentration over a 30 minute period.

Generation Rate

The mass concentration of the aerosol is measured by a gravimetric analysis technique. The aerosol is collected downstream of the photometer on a 47 mm diameter teflon filter (0.5 μm pore unbacked

Fluoropore filter, Millipore Corp., Bedford, Massachusetts 01730)* with the sampling flow rate controlled by a critical orifice at 10 L/min. The collection time was adjusted so that the aerosol sample collected was about 1 mg. This required 10 minutes for a mass concentration of 10 mg/m^3. Figure 12 shows measurements of the mass concentration of aerosol and the net photometer output covering the range of interest. Correlation between the two quantities appears linear. The photometer output thus provides a convenient method for monitoring the aerosol concentration and generator stability. The net photometer output in figure 12 was obtained with a 10 inch span Esterline recorder corrected for impedance mismatch and based on the difference of readings recorded with and without the aerosol. Figures 9, 10, and 11 show arbitrary units only. Other photometer/recorder systems may also be used provided that a gravimetric calibration is performed. The photometer assembly should be cleaned by directing a flow of clean air into the photometer chamber after two hours of generator operation.

Carbon Conversion Efficiency

As pointed out in the introduction, a quantity of prime importance in this study is the fuel to aerosol conversion efficiency, defined as the percentage of the carbon in the fuel converted to particulate matter. The following formula is useful for calculating the conversion efficiency, E:

$$E = 100 \text{ (mg aerosol generated per minute)/(mg carbon in fuel consumed per minute)}$$

$$= 100\, M_1 V_1/(M_2 V_2)$$

where

M_1 = mass concentration of aerosol in mg/m^3

V_1 = total aerosol flow in m^3/min; typically 0.030

M_2 = mg carbon in one cm^3 of fuel (0.98 for acetylene at 25° C and 1 atmosphere pressure (1.01 x 10^5 Pa))

V_2 = fuel flow rate in cm^3/min.

The average mass concentration of the aerosol for runs 2A and 2B (table 1 and curves in figure 11) was found to be 9.2 mg/m^3. Using this together with the fuel flow rate of 13 cm^3/min, one obtains a conversion efficiency of 2.2%.

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*Certain commercial equipment, instruments, or materials are identified in this paper in order to adequately specify the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that the material or equipment is necessarily the best available for the purpose.

A dominant characteristic of this carbonaceous aerosol generator, as well as others such as the premixed acetylene flat flame burner developed by Toossi [4], is the suddenness of the onset of sooting. Using a prototype combustion chamber, it was found that a 25% increase in fuel flow resulted in a 150 fold increase in aerosol output as monitored with an electrical aerosol analyzer. Because of this precipitous increase in aerosol concentration with fuel flow, it is not convenient to vary the soot concentration by simply varying the fuel flow. Instead, the mass concentration of the aerosol is varied over the range 5 to 10 mg/m^3 by changing the percentage of air in the air nitrogen mixture of the diffusion air. The lower the percent of O_2 the higher the aerosol concentration. Varying the dilution air may also be used to change the concentration.

EFFECT OF VARIOUS PARAMETERS ON THE GENERATION RATE

The effects of a number of parameters on the conversion efficiency were measured. These parameters included the type of fuel, the addition of various oxidizing agents to the fuel, diameter of burner tip, and the temperature of the fuel and air.

It was found that acetylene began sooting at a lower flow rate (40 cm^3/min) than the flow rate for propane (65 cm^3/min). The conversion efficiency is high under these conditions. For example, the conversion efficiency for acetylene is about 9% at a flow rate of 40 cm^3/min with a mass concentration of about 120 mg/m^3, which is about ten times higher than needed for inhalation experiments with animals.

As a sidelight, it was found that acetylene fuel at 30 cm^3/min without additive produced a high number concentration ($10^7/cm^3$) of very small aerosol particles, on the order of 0.01 μm diameter; however, the mass concentration of the aerosol was low, less than 0.1 mg/m^3. These small particles always seem to be produced when the fuel flow rate is below the sooting point. Detailed physical characteristics of flame-produced particles are presented in the next section.

With the addition of a small amount of premixed O_2, 3 to 4 cm^3/min, to the acetylene diffusion flame, a sudden increase in aerosol concentration and particle size was observed at fuel flow as low as 13 to 14 cm^3/min. The use of O_2 as an additive was motivated by a discussion with Y. Manheimer-Timnat from Technion, Haifa, Israel, in which he mentioned that the height of a flame at which sooting occurred greatly decreased with the addition of a small amount of O_2. Wright [5] has reported that the efficiency of conversion of fuel to particulate is greatly enhanced in ethylene and benzene with the addition of about 10 to 15% stoichiometric O_2. By using the O_2 additive in the fuel, the fuel flow rate can be reduced to a half or less of that required without the additive for a given soot generation rate. It was also found that the addition of N_2O had an effect similar to that of O_2 though a somewhat higher flow rate of N_2O is required compared to O_2.

The preheating of the fuel to about 100° C seemed to have a minor effect on the soot generation efficiency. The heating of the combustion chamber and diffusion air mixture tends to decrease sooting efficiency, though temperature control was found necessary to ensure generation stability.

The inside diameter of the burner nozzle was found to have little effect on the magnitude of the fuel flow rate at which sooting commenced. For ethylene fuel, changing the burner diameter from 1.8 to 8 mm resulted in only a 10% change in the fuel flow rate required for sooting. The onset of sooting was noted by the visible black stream rising from the flame.

While the size of the burner nozzle did not have a large effect on the sooting point, the thermal properties of the material used for making the tip were found to be important when using acetylene. Nozzles constructed of stainless steel tended to increase carbon deposition, which eventually resulted in clogging, while those made from brass did not have this problem. The selection of inside diameter for the nozzle (or flow velocity) was based mainly on the ease of fuel ignition by the high voltage electrode located at a given distance from the nozzle.

PHYSICAL AND CHEMICAL CHARACTERIZATION OF THE CARBONACEOUS AEROSOL

It is generally recognized that the particle size distribution is the most important quantity for determining the lung deposition characteristics of an aerosol. Particle measurement techniques used in the two studies of diesel particulate cited earlier were used in the present work [1] [2].

The primary instrument used for monitoring the particle size distribution was the electrical aerosol analyzer (EAA), which measures an effective particle size based on electrical mobility. The data are presented in terms of the particle number distribution ($\Delta N/\Delta \log D_p$) and particle volume distribution ($\Delta V/\Delta \log D_p$) versus D_p where N is the number concentration of aerosol per cubic centimeter. The basic data are the current corresponding to N versus voltage corresponding to D_p. An example of the output from the instrument is included in the top portion of figure 11 (the multipeaked curve) for eight discrete voltage settings. The size distributions are derived from the current versus voltage data using the sensitivity factors in Liu and Pui [6] for the case $Nt = 1 \times 10^7$ $(cm^3)(sec)$. A log-log plot is necessitated by the wide range in particle size and concentration. The quantities ΔN and ΔV refer to the number of aerosol particles in the particle diameter size range $\log D_p$ to $\log D_p + \Delta \log D_p$ and to the volume of aerosol particles in the particle size range $\log D_p$ to $\log D_p + \Delta \log D_p$, respectively. The size distributions (number and volume) are plotted in figures 13 and 14 for the case of the carbonaceous aerosol diluted in a 1.8 m^3 chamber. The undiluted aerosol stream has a mass concentration of 5.6 mg/m^3 (Run 1A).

It is convenient to characterize a size distribution in terms of the geometric mean diameter, $\bar{D}_g$, and the breadth of the size distribution by the geometric standard deviation, σ_g. These quantities are defined below for the number distribution:

$$\log \bar{D}_{gn} = \sum_{i=1}^{n} \frac{\Delta N_i \log D_i}{N},$$

$$\log \sigma_{gn} = \left[\sum_{i=1}^{n} \frac{(\log D_i - \log D_{gn})^2 \Delta N_i}{N} \right]^{1/2},$$

where N is the total number of particles and ΔN_i is the number of particles in the ith interval. A similar definition applies for the volume distribution except that N in the equations above is replaced with V.

Comparison of Diesel and Generator Aerosol

The parameters $\bar{D}_g$ and σ_g are listed in table 2 both for the generated carbonaceous aerosol and for the average diesel smoke for an Oliver/Waukesha F310 DBL six cylinder open diesel engine [1]. The parameters for the diesel smoke are based on particles in the size range of about 0.07 to 1 μm. It is seen that there is a good agreement for the geometric mean volume diameter for the diesel particulate and the carbonaceous aerosol from the generator.

The size distribution of the aerosol was found to be relatively independent of the combustion conditions in the generator provided that the total mass concentration of the soot was in the range of 3 to 15 mg/m^3. For example, in table 1 the value of $\bar{D}_{gn}$ changed by only about 0.01 μm as the total mass concentration of particulate increased from 5.6 (0.14 μm) to 9.1 mg/m^3 (0.15 μm). As mentioned in the previous section aerosol produced under a non-sooting condition has small particles, $\bar{D}_{gn}$ = 0.01. As a result, the mass concentration is low and number concentration is high. The number size distribution for this case is plotted in figure 15.

The second method of size analysis is based on aerodynamic size classification with the Anderson cascade impactor [8]. The aerodynamic particle size is the effective size for a unit density sphere. The concentration of particles on each stage was determined gravimetrically. In one experiment 98% of the carbonaceous aerosol by mass was found to be less than 1.1 μm aerodynamic diameter, 91% less than 0.7 μm, and 78% less than 0.4 μm. As in the case of the FAA, the results with the impactor were found to be similar for a range of generator conditions. For example, for three different experiments performed over a six week period the range in the percent of total particulate less than 0.4 μm varied from 79% to 86%.

As shown in table 2, there is reasonable agreement in the percent of aerosol less than 1.1 μm for the generator aerosol and the high temperature (> 300°C) exhaust from a 3150 caterpillar diesel engine [2]. At the lower exhaust temperature, there is a notably larger percentage (between 6 and 19%) that is greater than 1.1 μm in aerodynamic particle size.

The conventional cascade inertia impactors are not capable of detailed size classification of the aerosol of interest, because a major fraction of the aerosol is below the minimum sizing range of the impactor. A recently developed low pressure impactor [7] was used to measure the aerodynamic particle size down to 0.05 μm. Because of the low flow rate and small nozzle size, the samples collected were too small to be measured gravimetrically. Visual observation of the opacity of the deposited aerosol on the collection surface indicated that the major fraction of the aerosol was deposited on the last two stages, corresponding to particles sizes of 0.075 to 0.05 μm. This was found to be the case for both coated (silicone stopcock grease) and uncoated collection surfaces. By comparing the opacity of the last two stages, it was found that the major deposition was on stage 7 (0.075 μm).

The third technique for particle sizing was electron microscopy. The samples were collected on microscope cover slides and on 2.3 mm diameter copper specimen grids with a carbon coating using a Thermo-System electrostatic precipitator. Preliminary work with a scanning electron microscope indicated that the particles had an agglomerate type structure not unlike those produced by the carbon black industries [9] but the resolution was not adequate for analyzing detailed structure. As shown in figures 16 and 17, transmission electron microscopy clearly indicates that the aerosol particles are made up of an agglomeration of nearly spherical elementary particles of about 0.023 μm in diameter. There is qualitative similarity between this picture and those presented in the diesel study by Vuk et al. Their rather extensive particle analysis showed that the mean elementary particle size based on number is 0.026 μm. The mean size refers to the particle size at the 50% point; that is, half of the number of particles are larger and half smaller than the mean. The elementary particle size for the generator was found to be about 0.023 μm based on sizing 108 particles. As a measure of the agglomerate particle size, the hypothetical diameter of the largest sphere which can cover the entire agglomerate was used. The mean largest sphere diameter, 0.54 μm, given in table 2 is seen to far exceed the particle size measured by the other methods. Vuk et al. found for diesel particulates that the largest sphere diameter exceeds the aerodynamic particle size by a large amount just as in the case for the carbonaceous aerosol from this generator. Discussion of measurement error is given in Appendix 2.

Elemental analysis of the aerosol collected showed a carbon-hydrogen ratio of 99 to 1 or more for carbon. The analysis was performed by a commercial contractor (Schwarkopf Microanalytical) on about 4 mg

particulate samples on glass fiber filter by measuring the amount of CO_2 and H_2O produced by the combustion of the samples. Results from duplicate samples agreed within 0.5%. As shown in table ?, the diesel particulate at high exhaust temperature was found to have almost as high a carbon content, 98%. At lower exhaust temperatures, the percent carbon drops, presumably because of an increased amount of condensed hydrocarbons [2].

Aerosol Size and Repeatability

Table 1 shows the consistency in the concentration and geometric mean size of some sample carbonaceous aerosols obtained in this study. The results are given for two concentrations (1 and 2) with duplicate measurements and a third measurement to determine the effect of coagulation. All results except for the mass concentration were obtained using the electrical aerosol analyzer. Each experiment required 30-40 minute operation of the generator to allow time for the collection of two filter samples (10 to 15 minutes each) and for time to measure the size distribution with the electrical aerosol analyzer. The necessary dilution of the aerosol for operation of the EAA was obtained by directing the aerosol into a 1.8 m^3 dilution chamber for a known period of time (23 minutes). The results indicate that the generator has the stability and repeatable aerosol size characteristic for animal exposure study in a flow through system.

Effect of Particle Coagulation

The third experiment showed the effect of aging on the concentration and size distribution. As the particles collide and coagulate due to Brownian motion, the number concentration will decrease and the particle size will increase. This process does not affect the total mass of the suspended particles but it does affect the number concentration and particle size. That coagulation as an important aging mechanism is clear from the results in table 1 (run 3) where it is seen that the number concentration drops by 20% while the volume (mass) concentration remains about the same after 32 minutes. It is also seen in table 1 that there is a slight increase in the mean particle size.

The fundamental quantity for coagulation is the coagulation coefficient, Γ, which is defined by

$$dN/dt = \Gamma N^2$$

From this equation and the data in table 1, one finds that $\Gamma = 4.1 \times 10^{-10}$ cm^3/s. As defined above Γ is an average coagulation coefficient, though in reality Γ depends on particle size, charge, and perhaps on shape. Because of the relatively high concentration of the carbonaceous aerosol, about 3×10^6 particles/cm^3, the coagulation effect may be important in affecting the particle size if the generator output is run into an accumulator for some length of time before entering the animal chamber. At this concentration, the particle size will increase by about 10% in five minutes.

Gas Analysis

An analysis of the CO and CO_2 in the aerosol stream was made because of the potential interference of the C^{14} in gaseous form with the particulate analysis. Using a long-path-length infrared gas cell, it was found that the concentrations of CO_2 and CO were 840 ppm and 10 ppm respectively for the case of an acetylene flow rate of about 14 cc/min. From a carbon balance, one finds that about 90% of the carbon in the fuel is converted to CO_2, 1% to CO and the remainder to particulate.

SECTION 5

DISCUSSION

The prototype generator developed appears to be well suited for lung deposition studies because of its constant output and consistent particle size distribution. However, the conversion efficiency of the generator may still be too low for long-term economical use with a radioactive tagged fuel. There are a couple of alternative generator designs that might yield higher efficiency. One method is to use liquid aromatic fuels such as benzene or toluene. These fuels have been shown to have a high conversion efficiency to particulate [5]. Another possible method widely used in the carbon black industry [9] is to generate the particulate by the thermal decomposition of the fuel in the absence of oxygen. Professor Flagan at California Institute of Technology has a tube furnace that might be available for making a preliminary study of the feasibility of such a technique.

A second area where additional research is needed concerns the characterization of the size distribution of the carbonaceous aerosol. For agglomerate type particles such as the carbonaceous aerosol, there is no theory for relating the particle size parameter of one instrument based on one aerosol property such as the electrical mobility, to the particle size based on another property such as aerodynamic size classification. In table 2 it is seen that the aerodynamic particle size from the low pressure impactor is a factor 4 smaller than the electrical mobility volume particle size. This difference is not simply an instrument calibration effect, because good agreement was found between the low pressure impactor and the EAA in the 0.05 to 0.1 μm diameter size range by Hering et al [7] using spherical sulfuric acid aerosol.

Even though the low pressure impactor results are qualitative at this point, the magnitude of the discrepancy with other methods justifies a carefully designed study with this newly developed measurement method. Quantitative results could be obtained even with the small amount of particulate deposited in each impaction stage by using C^{14} labeled fuel and sensitive radiation detectors. This method has the advantage over the other two techniques for particle deposition studies that it measures the aerodynamic particle size, which is known to be an important parameter for particle deposition in animals [10].

The low-pressure impactor by itself will not answer all the questions regarding particle deposition. The diffusion of particles in the respiratory tract passages becomes an important mechanism of deposition for sufficiently small particle sizes. In a study of aerosol deposition at the bifurcation of airways under conditions simulating flow in the lung, Bell [11, 12] found that the major mechanism of aerosol deposition for spherical particles less than 0.1 μm in diameter is diffusion. Over the size range from 0.1 μm to 1 μm there is a transition from diffusional to inertial deposition (impaction). A proposed deposition and retention model for aerosol

in the respiratory tract developed by a special task group on lung dynamics set up by Committee II of the International Commission of Radiological Protection [13,10] indicates that a significant fraction of particles smaller than 0.1 μm diameter are deposited by diffusion in the pulmonary and T-bronchial region of the lungs.

These studies suggest that diffusional deposition might be an important mechanism for the deposition of carbonaceous aerosols in the lungs. It is not possible to predict the diffusion coefficient of the carbonaceous aerosol, because the relation between particle diffusion coefficient and the aerodynamic diameter is unknown for cluster and chain type agglomerates. Therefore, we suggest that the diffusion coefficient of the carbonaceous aerosol be determined. This can be done by using a diffusion battery together with a condensation nuclei counter.

While the particle size of the aerosols from diesel exhaust and from the generator are similar, the morphology (including shape and surface area) of the particulates has not been compared in the present work.

SECTION 6

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APPENDIX A

DETAILED CONSTRUCTION & OPERATION OF THE GENERATOR

Construction and Function of Parts in the Combustion Chamber

Body

The body of the generator (figure 1) consisted of the top and bottom chambers jointed together by bolts and sealed by two(2) copper gaskets at the mid section. Each gasket is welded to one of the porous cylinders. In addition to the main chambers, there are the exit and burner sections. These sections are also sealed to the main chamber by use of copper gaskets and bolts. Each of the 4 stainless steel sections may be disassembled. Both the exaust nozzle and burner nozzle can also be removed.

Heaters and Thermocouples

Eight 40-watt heaters, connected electrically in parallel, are embedded in the two(2) chambers. There are three type K thermocouples: the one located in the bottom chamber is connected to the temperature controller; the one at the exit section extends into the exit nozzle, the third one is for monitoring the body temperature of the top chamber.

Air Mixture and Fuel Inlets

The diffusion air mixture is distributed by a swagelock tee to the top and bottom section of the chamber. The gas is heated as it travels through the chamber and diffuses through the porous metallic cylinder. Each chamber can be independently supplied by different mixtures. A flame arrestor separates the fuel inlet and the downstream fuel line.

High Voltage Ignition and Pressure Monitor.

The burner section contains a H.V. ignition lead and an opening for monitoring the chamber pressure. The high voltage terminal is insulated from the chamber by a ceramic tubing. It should be protected from any impact because of its brittleness. Disconnect or connect the lead carefully by pulling straight down or inserting straight up thru the tygon tubing. Any arcing from H.V. lead to chamber ground would prevent any firing at the nozzle.

Connections

All connections to the chamber should be made carefully according to line labels. Fuel and air mixture lines should be identified using odor from C_2H_2 or by other means after initiating free flow and before the final connection.

Connect the ground wire from the power line to the chamber at one of the body bolts at the midsection of the chamber. The bolt may be loosened and retightened by a proper allen wrench.

Control Panel Functions

Line Connections

Both the inlet and outlet gas lines are equipped with Quick Disconnect joints located at the side of the control cabinet and identified by labels. Both the male and female joints contain valves which shut off the flow after the joint is disconnected.

Gas and Power requirement:

Power: 115 VAC, 10 amp.
Air : 10-15 psi, 30 L/min, filtered
O_2 : Bottle, regulated 15 psi, 10 cm^3/min
N_2 : Bottle, regulated 15 psi, 1 L/min
C_2H_2 : Bottle, regulated (do not exceed) 10 psi, 20 cm^3/min

Gas Flow Meter and Control

After entering the inlet at the cabinet the gas goes thru the in-line filters, an on-off toggle valve, a needle control valve, and finally the rotameters. Dilution air flow is controlled by a needle valve and monitored by the magnehelic gauge. Because of high flow volume, a supplemental and disposable filter should be used upstream of the air inlet.

Calibration curves for each flowmeter are given in the text. A bubble flowmeter provided with the instrument may be connected at a specific outlet to check or recalibrate the flowmeter for a specific gas. Once the needle values for the fuel and O_2 additive are set, avoid changing the settings.

Temperature Controller and Indicators

A time proportional temperature controller, though designed for type J thermocouple and Fahrenheit scale, will maintain the chamber about 40°C at a setting of 90. The temperature is displayed by one of the digital temperature monitor. Controller setting does not correspond exactly to the desired chamber temperature because the required type J thermocouple was not used. The digital indicator using Type K thermocouple is correct, however.

The exhaust gas temperature is monitored by the other digital indicator which is connected to the audible alarm. The alarm will trigger when exhaust temperature drops below 50°C. Other trigger temperature may also be selected by connecting the proper channels of the BCD output at the back of the meter.

Safety Alarm and Photohelic Gauge

The purpose of the electrical audible alarm is to warn the operator of incorrect pressure conditions in the chamber or flame-out situation. The alarm is activated by the indicated toggle switch at the top part of the panel. The alarm should be turned on when the generator begins to operate after the initial ignition and start-up. The center magnehelic gauge monitoring the chamber pressure is connected to the alarm. Adjustment knobs at the front of the gauge will bracket the desired safe operating pressure. Pressure below or in excess of the set points will trigger the alarm. It is a good practice to move the set points into alarm position and then back off slightly after the chamber is warmed up and operating properly.

Magnehelic Gauge Manometer

In addition to the magnehelic gauge which monitors safe operating pressure, there are two other gauges. One manometer measures the chamber pressure. Any leakage, malfunction of gas flow rate, or flame-out will be indicated. The other gauge monitors the orifice meter which measures flow rate of dilution air. At a pressure drop of 0.68 cm H_2O (66 Pa), the flow rate is 27 L/min as shown by the calibration curve.

High Voltage Ignition

A Fenwal direct spark ignition (-05-14) device is used for fuel ignition at the burner. A momentary contact button (red) when held at on-position will initiate and maintain the high voltage to the ignitor inside the chamber. Once ignition is confirmed, the button should be released. Full ignition is indicated when the exhaust temperature indicator begins to climb above 70°C and stay at this value. Special flow rates for N_2 and air are needed to start the fuel ignition (see operating section).

Monitoring Photometer

The monitoring light-scattering photometer contains an ESL (Electro-Signal Lab) photoelectric smoke detector with analog output from the photodiode. The input connector (amphenol) has 4 lines: 2 for the 6 volt AC to power the light source and the electronic components, 2 for the signal on light scattering intensity.

The photometer has a flow through system with 3/4" OD copper tube inlet and outlet. The cover may be easily removed for cleaning. Lamp alignment in the photometer is critical in assuring that the calibration is valid. Jarring should be avoided. Recalibration based on light scattering and filter collection of the aerosol should be performed if misalignment is suspected.

Deviation from the normal base line output of the photometer when

exposed to clean air is an indication of the need for cleaning. For cleaning, the top cover should be removed and compressed air jet stream be directed at various points to dislodge the deposit.

Hook-Up Procedures

Electrical and Gas Lines

It is essential that the laboratory receptacle for the AC plug of the generator is polarized in accordance to standard electrical code. All electrical lines and gas tubings are labeled and should be matched in completing the connections between the chamber. Inlet tubings from bottled gases, especially O_2 and C_2H_2, should be installed and tested carefully.
Slight height adjustment by shimming the cabinet may be needed in aligning the rigid copper tubing from the cabinet to the mixing tee section of the combustion chamber.

Compressed Gas Bottles

Air - Filtered and pressure regulated laboratory air from compressed air line at 30 L/min may be used.

N_2 - Compressed regulator-controlled nitrogen in 1A bottle is recommended.

O_2 or C_2H_2 - Because of very small flow volume needed small bottles 5A and 6A with 2 stage regulator are recommended.

The distance between the gas bottles and the control cabinet should be short to avoid long time necessary for purging the line during start-up operation because of the low flow rates.

Operation

Procedures

After all the connections between the gas sources, control cabinet and combustion burner are made and checked, the following procedure is suggested for the start-up operation.

The output of the generator should be directed into a well-ventilated laboratory exhaust system.

Turn knob of controller to a setting of 90 for heating of the chamber.

Turn on dilution air and adjust to 27 L/min.

Turn air on at the toggle switch. Adjust flow rate to a scale reading of 50.

Turn on acetylene from compressed gas bottle, adjust regulator to about 10 psi, open toggle valve at the control panel for C_2H_2. Allow gas to flow for at least 15 minutes, or sufficient time to completely purge the long line. Do not adjust needle valve. Flow rate should be about 15 cm^3/min.

Turn on O_2 from bottle and adjust regulator to 15 psi and open toggle switch at panel and purge the O_2 line. Do not adjust the needle valve.

If the pressure in the chamber is above 0.3 cm H_2O (29 Pa) and odor in the exhaust nozzle shows presence of C_2H_2 (before diluting) the burner is ready for ignition.

Note the chamber and nozzle exhaust temperatures at the indicators. Turn ignition power switch on and alarm power off.

Push red button to initiate ignition. Release button if nozzle temperature has reached a high and steady value. Several attempts may be needed.

Once ignition is confirmed, adjust the gas flow rates to the following values (scale reading) for soot generation at 10 mg/m^3:

Gas	C_2H_2	O_2	N_2	Air	Dilution Air
Float Scale	G	G	SS	G	
Reading	22	27	85	45	0.68 cm H_2O
Pressure Psig	<10	15	15	10-15	10-15

Air may be varied from 55 to 45 in order to vary soot concentration from 3 to about 10 mg/m^3.

Safety Precaution

Acetylene has a wide flammable and explosive limit (2.5 to 82%). Standard procedure for handling explosive gas should be observed. No copper tubing should be in contact with the acetylene. Flame arrestor should be placed in series at the outlet, downstream of the pressure regulator. All tubing to the generator inlet should be less than 6 mm OD.

Line should be checked for leak after initial connection. Purging of the lines is necessary after an overnight shut down. Do not ignite until line is completely purged. Purging also assures that the regulator has reached equilibrium.

To shut the system down, simply decrease the airflow and increase the N_2 flow rate. Shut off all toggle valves after nozzle temperature indicator has confirmed a condition of flame out. Shut off valves at the cylinder head. Do not adjust the regulator valves.

Diffusion air must be flowing in the chamber before attempting ignition.

Leads to the H.V. ignition terminal has about 1200 volts. Do not use other leads and take necessary precautions to avoid shock.

Trouble Shooting and Maintenance

The exit section of the chamber may be removed easily in order to examine the condition in the burner section by sighting with a flashlight.

If the burner requires cleaning, remove all connecting lines and tubings before removing the burner section. In pulling the section out, be careful not to damage the insulating ceramic tubing covering the HV wire.

The generator has been in use for sometime at NBS. No foreseeable problems were encountered. If any unforeseeable problems arise, please call the authors at NBS.

APPENDIX B

MEASUREMENT ERRORS USING ELECTRON MICROSCOPY TECHNIQUE

There are a number of considerations that go into the determination of the accuracy of a size distribution measurement by electron microscopy including the resolution of the microscope, the analysis of the electron micrographs for particle size, the statistical significance of the sample size, and the nature of the collection process. Here we present a brief discussion of these factors and how they affect the accuracy of the particle size measurements.

The magnification of the transmission electron microscope (TEM) was determined by measuring the line separation of an optical spectroscopy grating with 2160 lines per millimeter versus the intermediate lens currents of the TEM. The resulting calibration curve has a relative error of up to $\pm$ 10%.

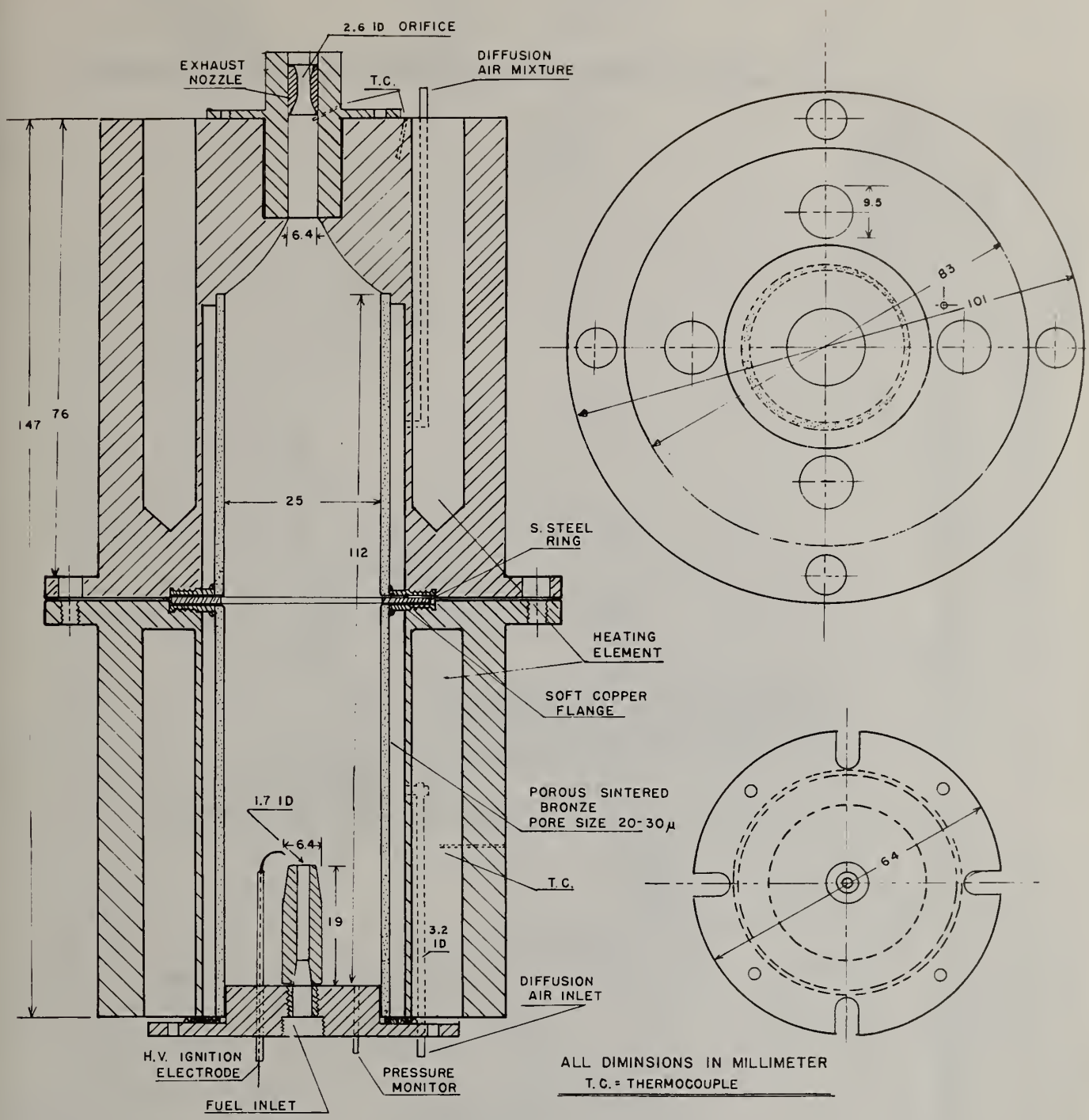
The actual particle size measurements were made on the prints from the microscope negatives by using a micrometer. The micrometer is graduated to 0.025 mm (0.001 inch). For example, a primary particle with a diameter of 0.023 μm would correspond to about 3.1 mm on the print, a magnification of 135,000.

The estimated error in the resolution of the micrometer is only a few percent compared to the estimated error of $\pm$ 10% associated with the uncertainty in determining the exact edge of the particle on the print.

To accurately determine the size distribution of a polydisperse particles, a large number of particle must be sized. In their TEM analysis of diesel exhaust particulate, Vuk et al sized 600 primary particles to obtain an accurate size distribution. In this study 100 primary particles were sized; this represents a large enough size to get a good measure of the mean particle size but only a qualitative estimate of the geometric standard deviation, σ_g.

Finally, the sampling of the carbonaceous aerosol into the copper specimen grids by the electrostatic precipitator may affect the size distribution. The shape of the deposited particle may be affected by the charging and the collection process under a high electric field. there may also be enhanced deposition of particles upon previously deposited particles because of electrical effects. Such effects would be significant in regard to the overall agglomerate size (greatest sphere diameter) but probably would have little effect on the primary particle size.

Taking into consideration the errors mentioned above, the uncertainty in particle size measurement using TEM is estimated to be $\pm$ 20-30%.



COMBUSTION CHAMBER FOR SOOT GENERATOR

Figure 1 Cross-sectional view of the combustion chamber

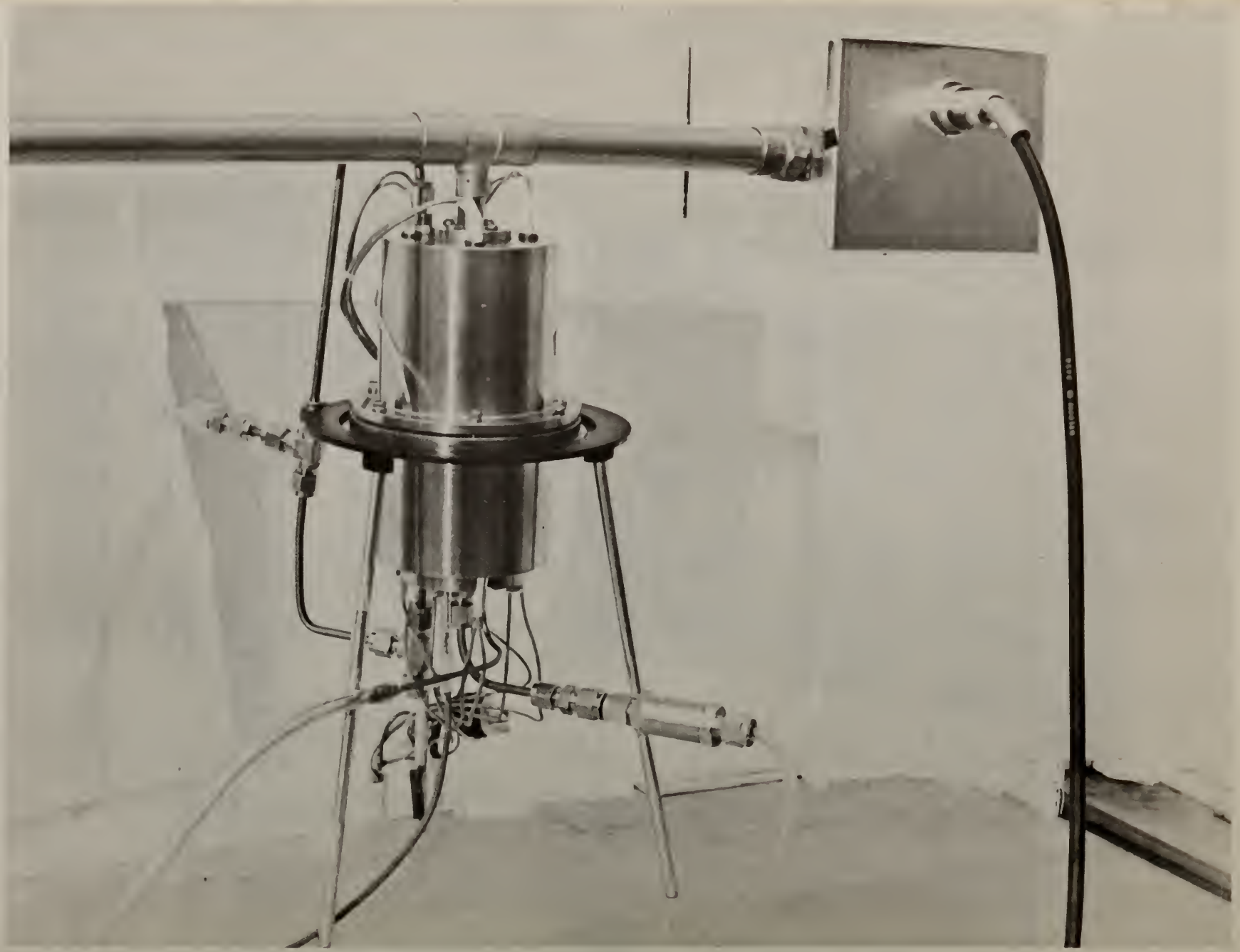


Figure 2 Combustion chamber and connections

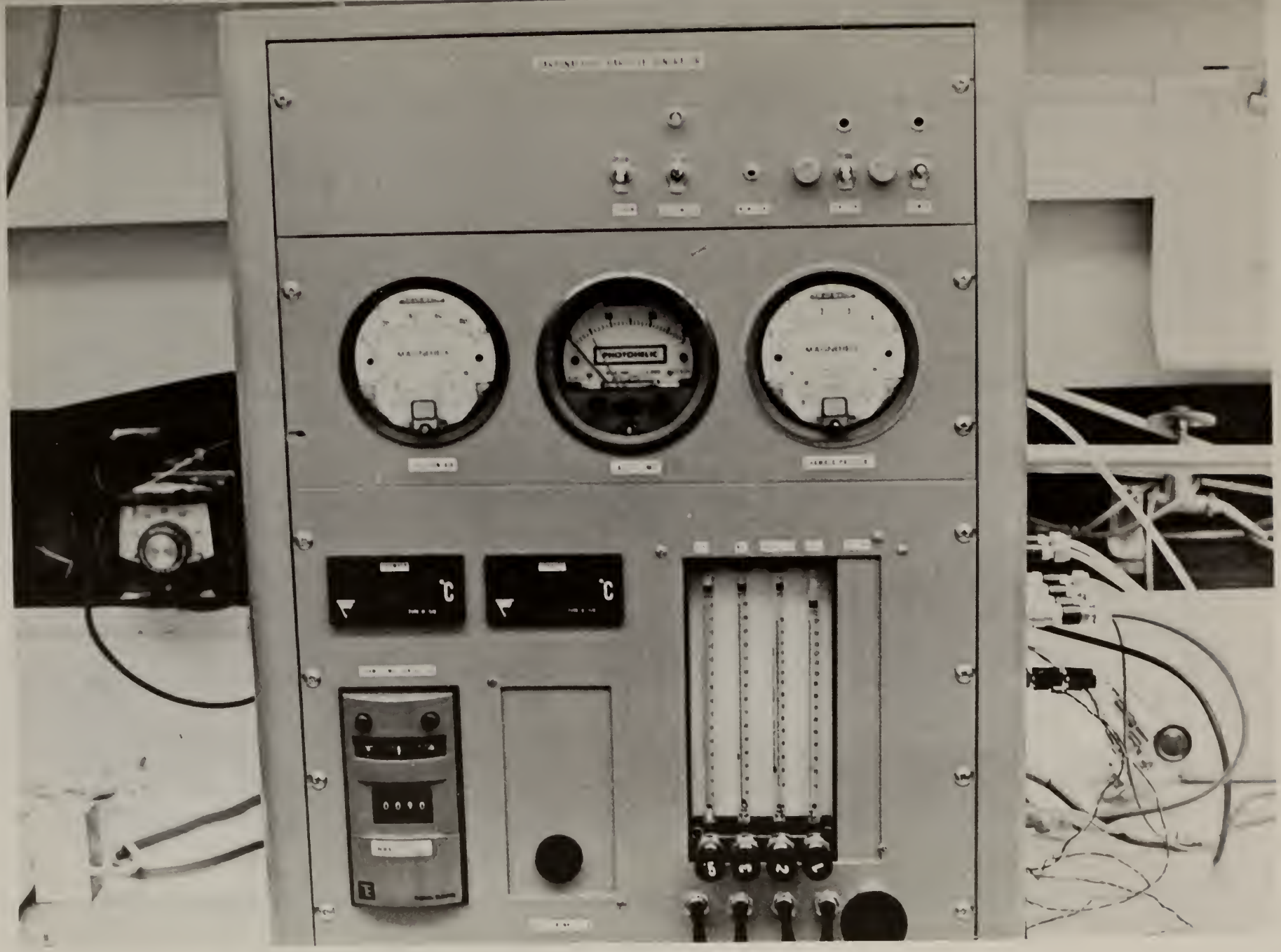


Figure 3. Control panel of the generator

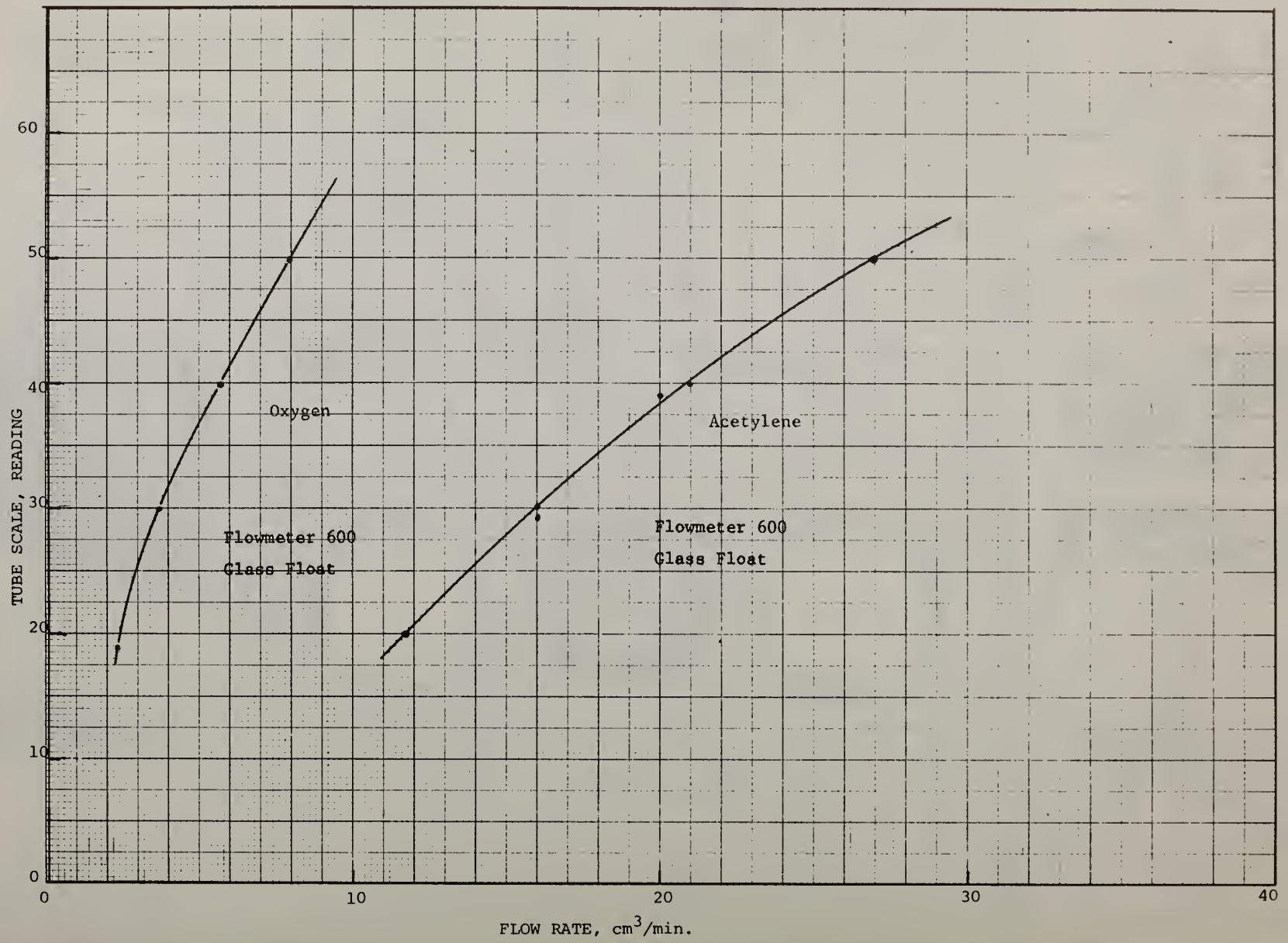


Figure 4 Calibration curves for acetylene and oxygen flow meters

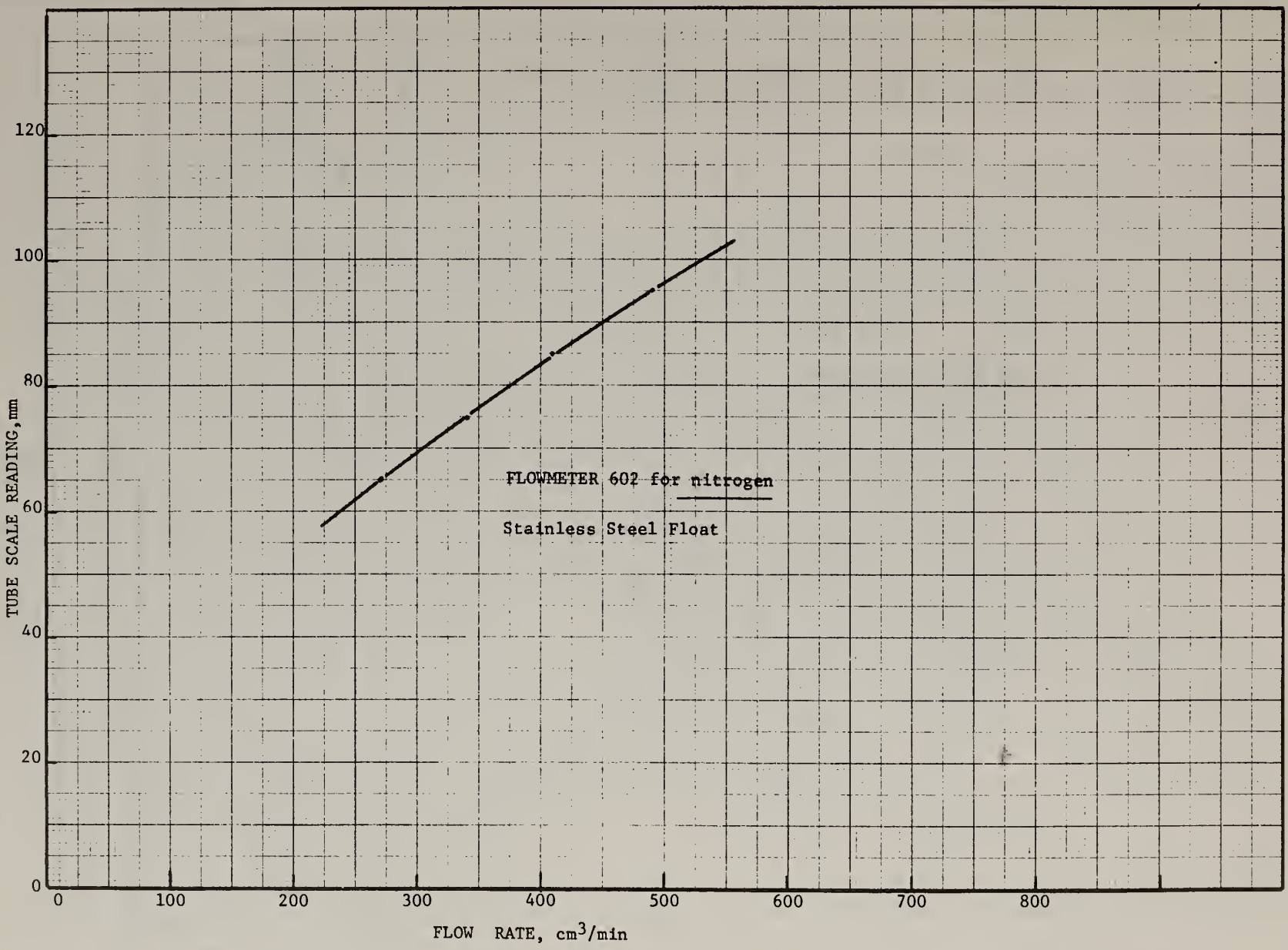


Figure 5 Calibration curve for nitrogen flow meter

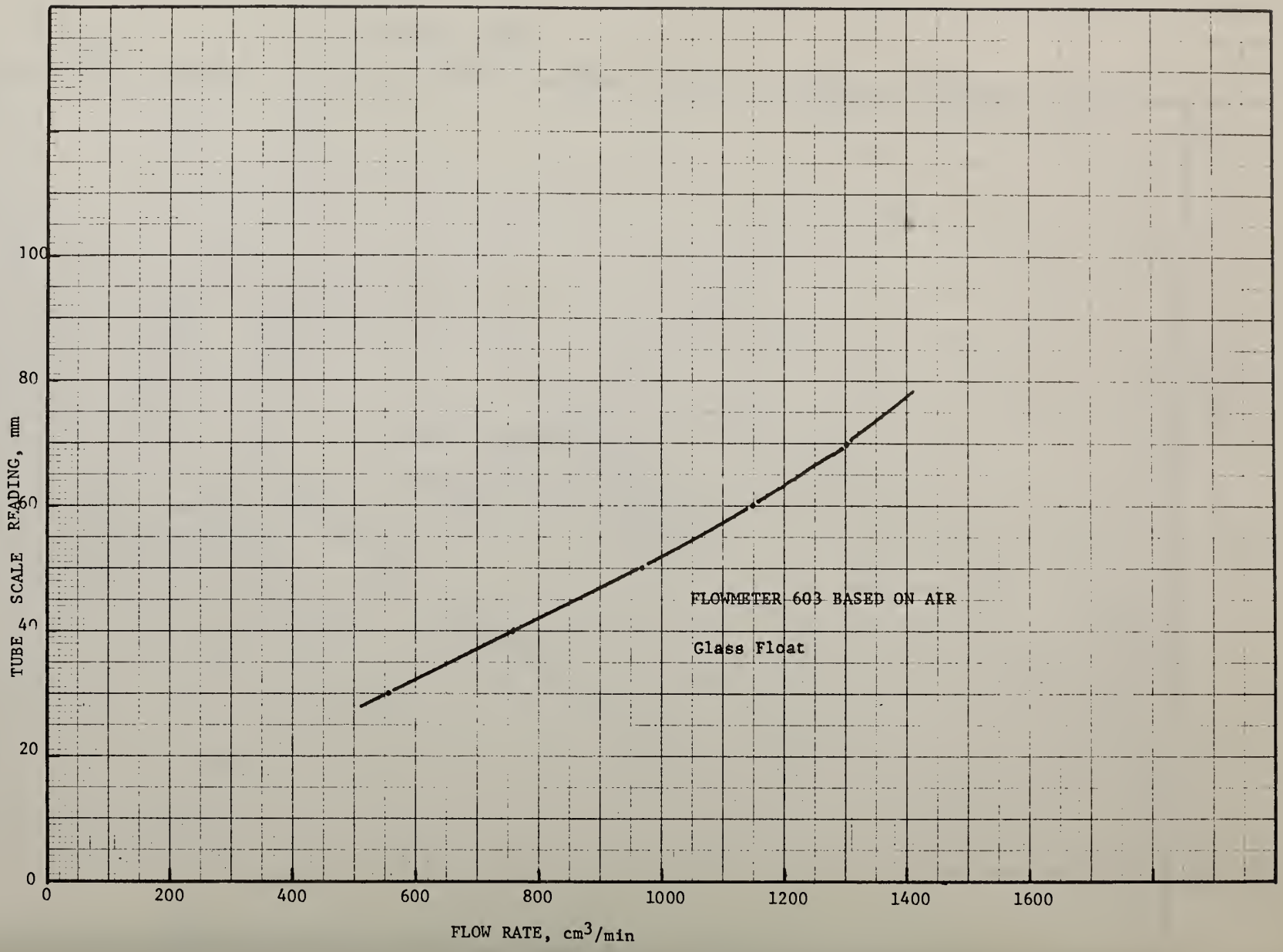


Figure 6 Calibration curve for air flow meter

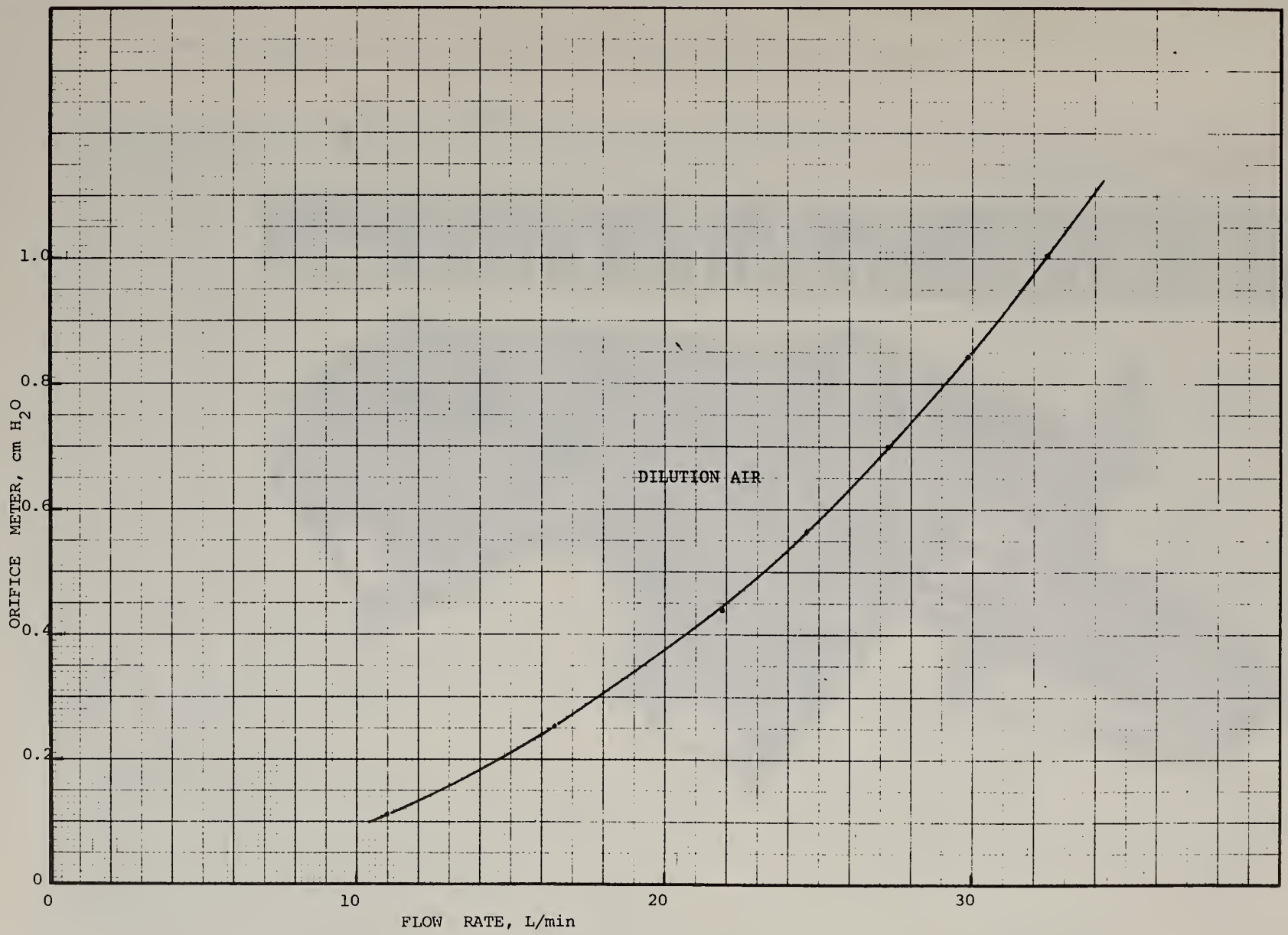


Figure 7 Calibration curve for dilution air gauge

Figure 8 Cross-sectional view of the light-scattering photometer

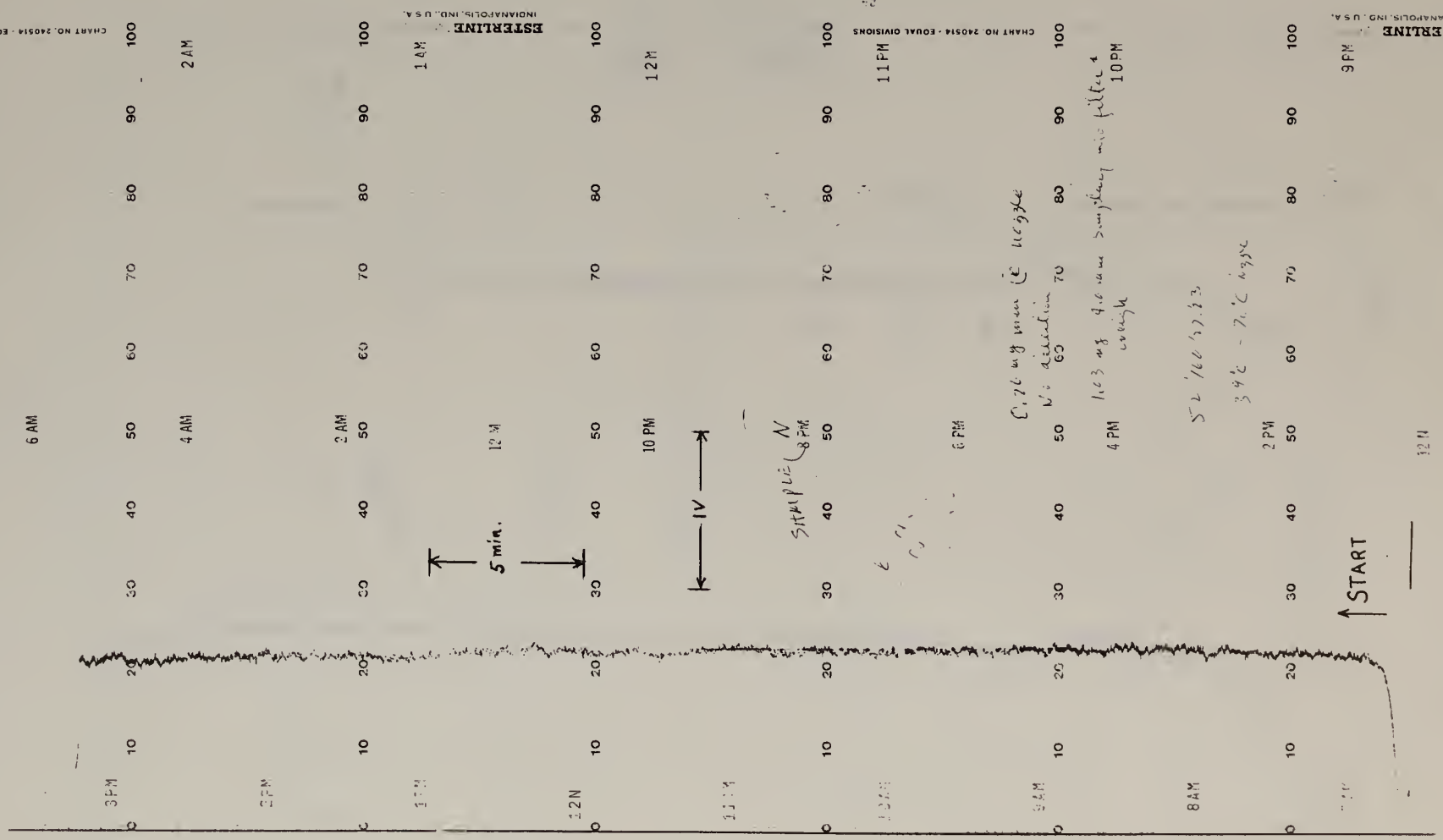


Figure 9 Output stability of the soot generator at 6.5 mg/m^3

Figure 10 Repeatability of the generation rate with on and off cycling of the generator

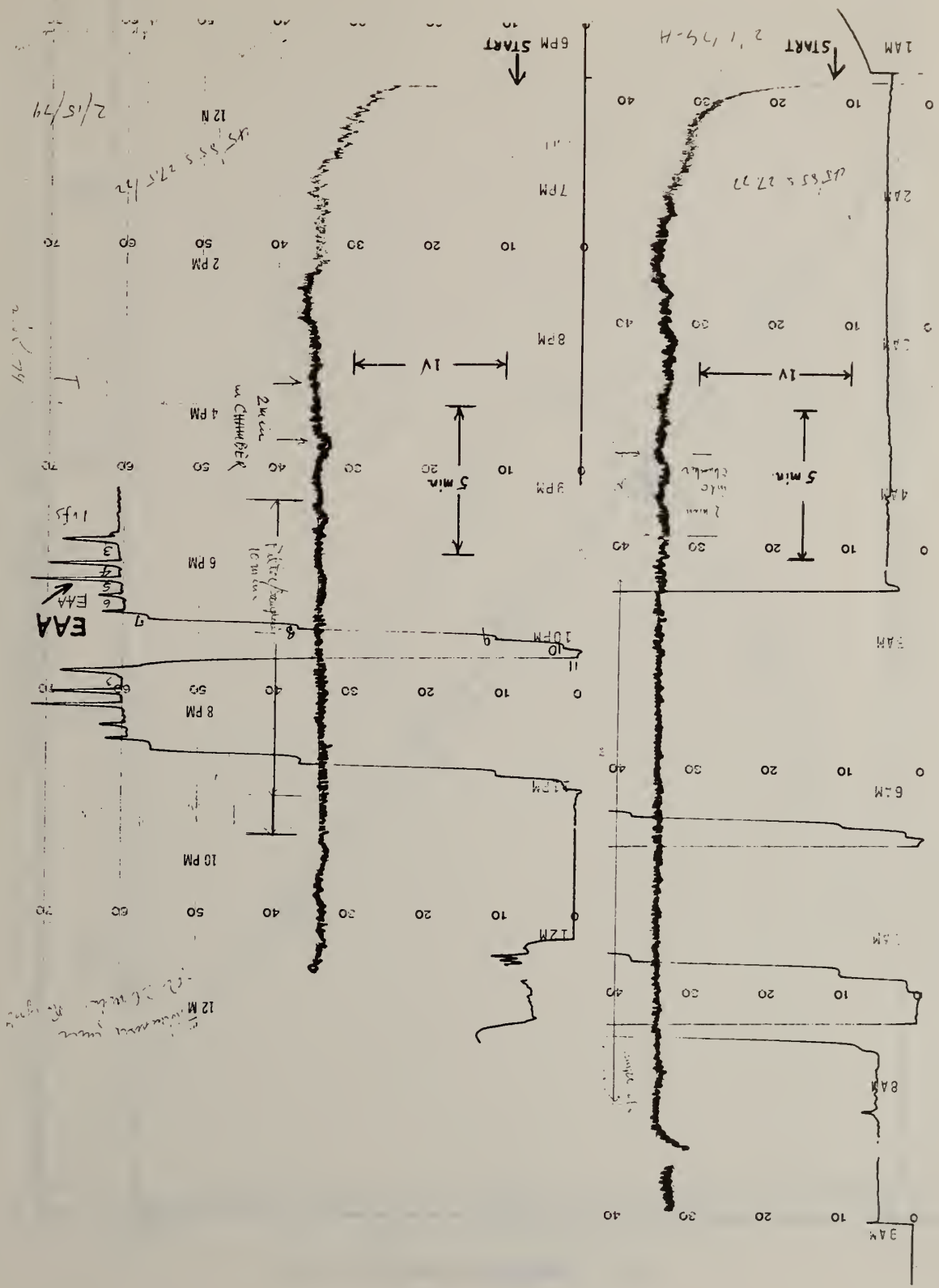


Figure 11 Comparison of runs 2A and 2B at high concentration levels

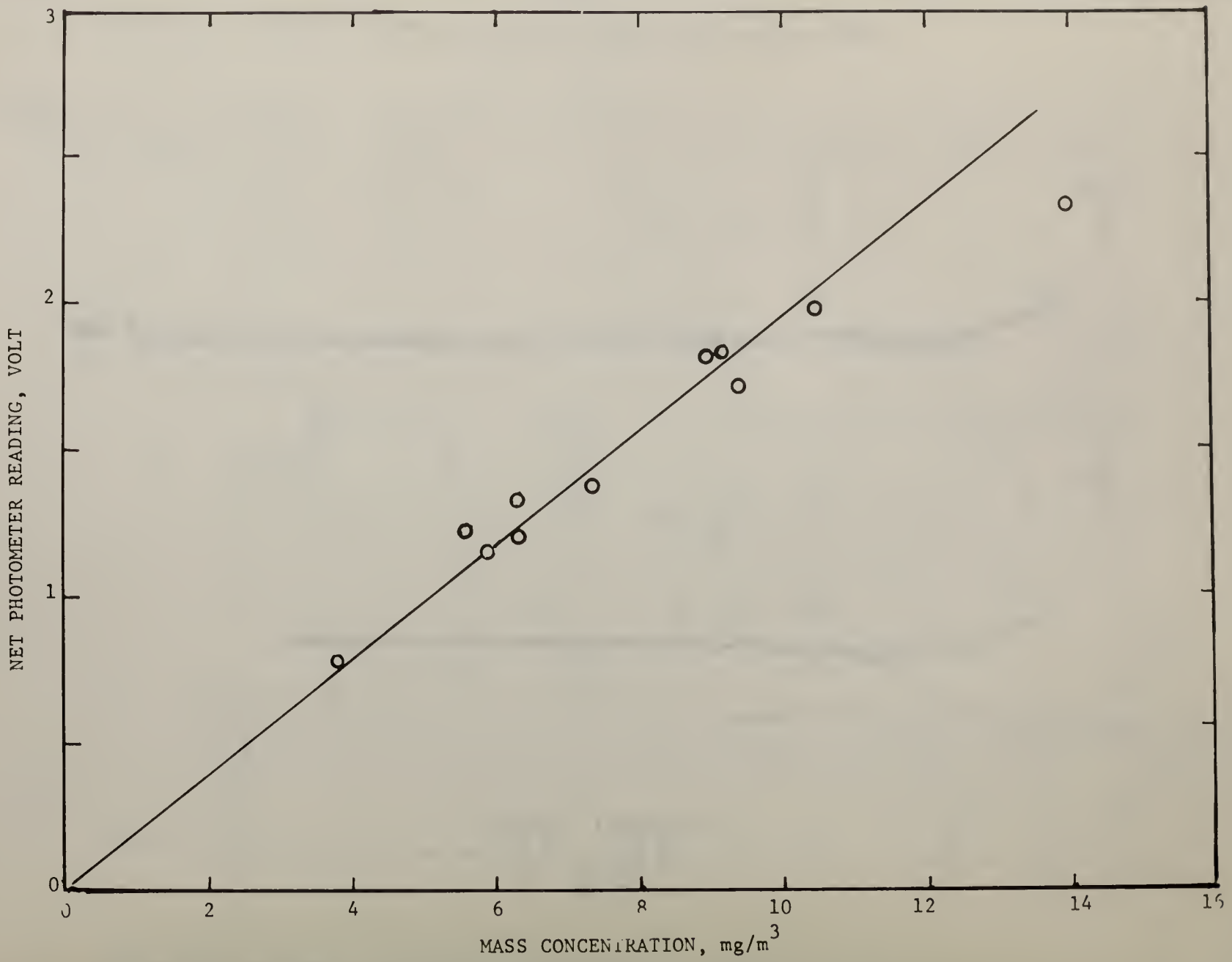


Figure 12. Photometer reading as function of mass concentration of the aerosol.

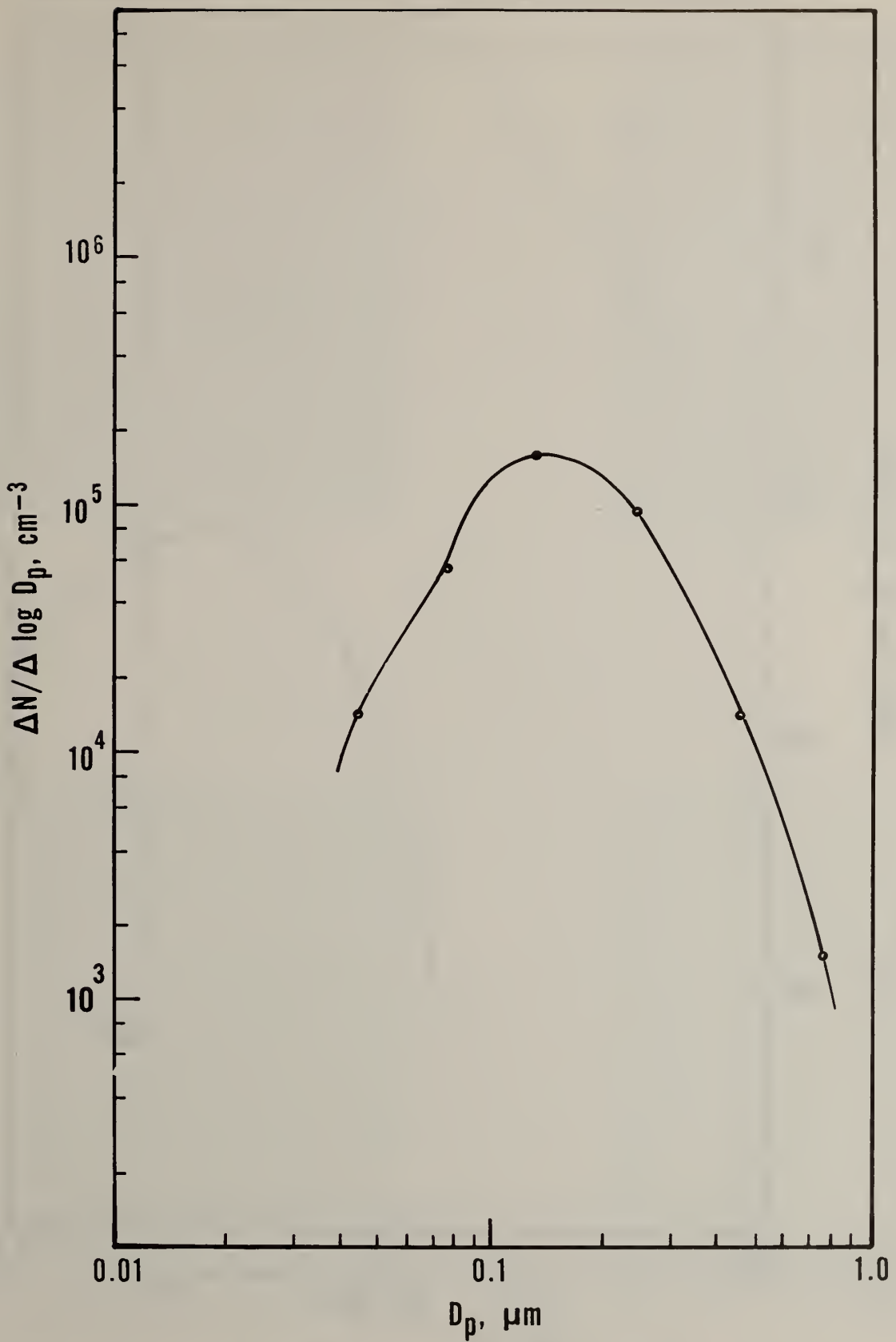


Figure 13 Typical number-size distribution of generated aerosol after dilution (30:1) based on EAA measurements

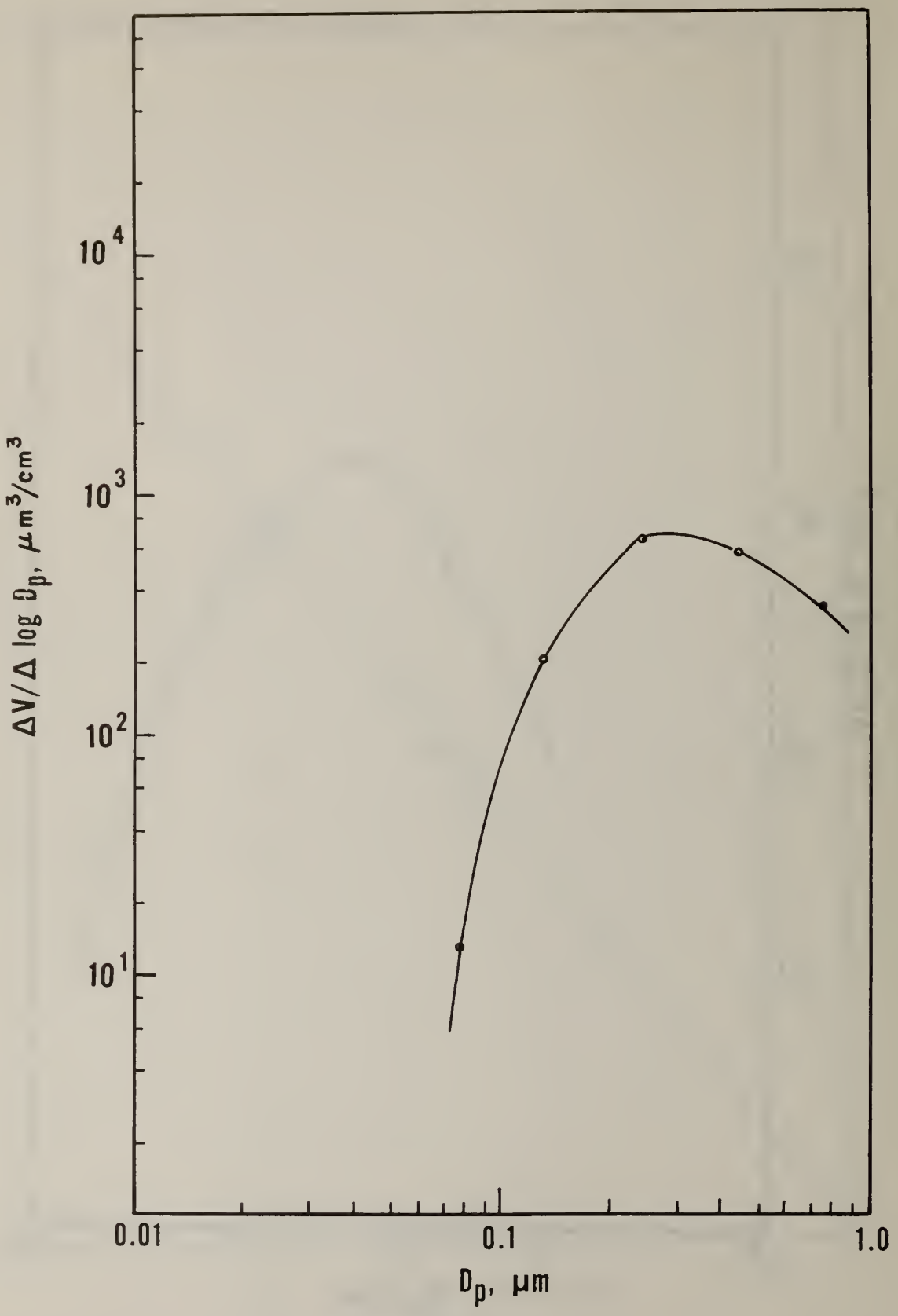


Figure 14 Volume-size distribution on the diluted aerosol based on data given in figure 13

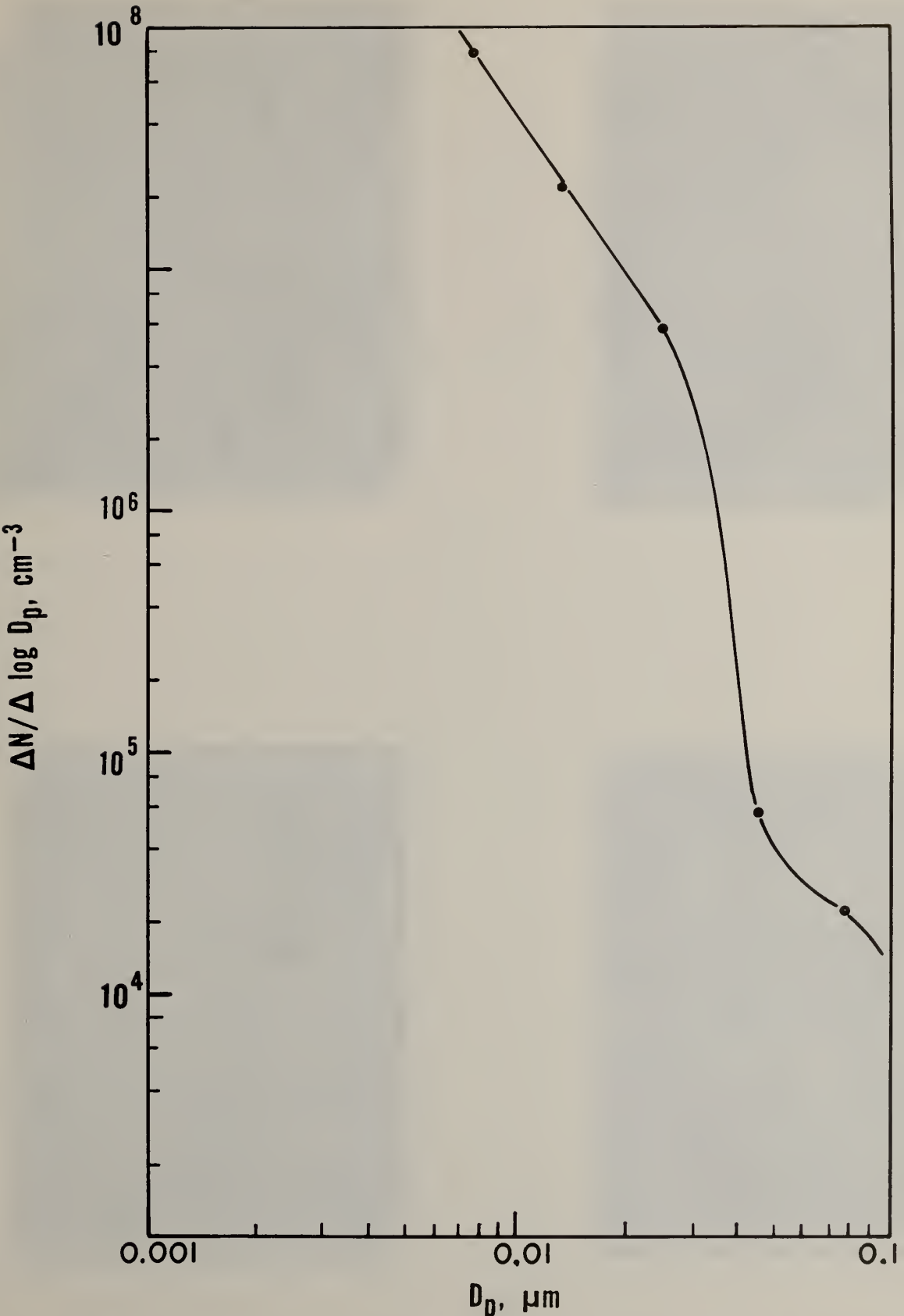


Figure 15 Number size distribution of aerosol based on EAA measurement from flame burning under non-sooting condition

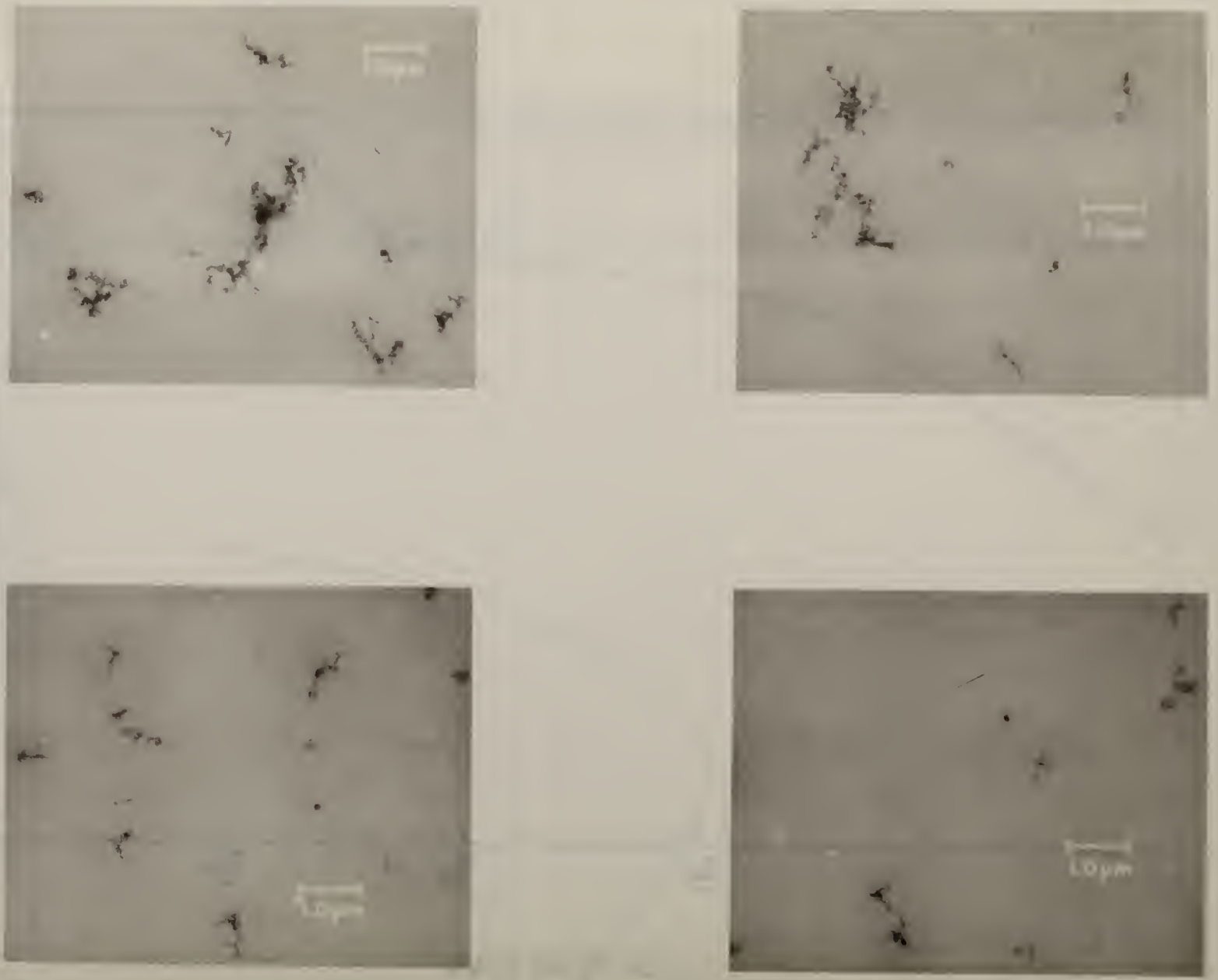


Figure 16 Photomicrographs of carbonaceous aerosol under lower magnification

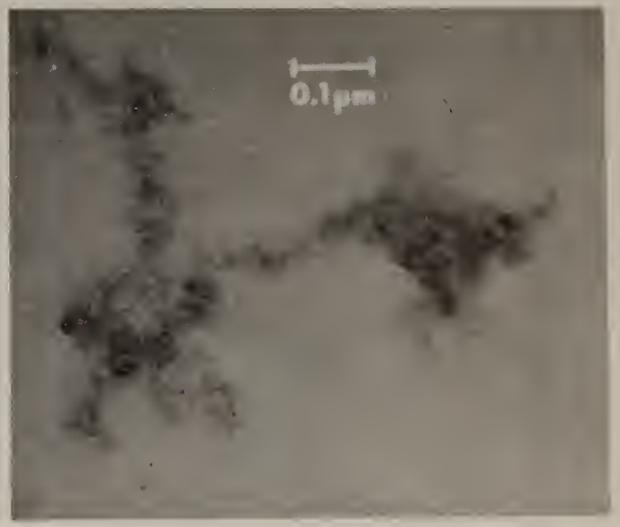


Figure 17 Photomicrographs of carbonaceous aerosol under high magnification

Table 1 Properties of Carbonaceous Aerosol[a]

Run #	Concentration Number $1/cm^3$	Volume $\mu m^3/cm^3$	Mass mg/m^3	Apparent Density g/cm^3	$\bar{D}_{gn}$ μm	σ_{gn}	$\bar{D}_{gv}$ μm	σ_{gv}
1 A	2.58×10^6	1.31×10^4	5.6	0.43	0.14	1.7	0.33	1.7
1 B	2.61	1.33	5.9	0.44	0.14	1.7	0.33	1.7
2 A	3.30	2.04	9.1	0.45	0.15	1.7	0.34	1.7
2 B	3.64	2.07	9.3	0.45	0.15	1.7	0.33	1.7
3 (t = 0)	0.285	0.152			0.15	1.6	0.34	1.8
3 (t = 1920s)	0.233	0.155			0.16	1.6	0.36	1.8

[a]All quantities except for the mass concentration were calculated based on data from the electrical aerosol analyzer. Concentrations in run 1 - 2 are given in terms of the original discharge concentration although measurements were made after the aerosols were diluted in a 1.8 m^3 chamber. Concentrations for run 3 are the chamber concentrations

Table 2 Comparison of Diesel and Generator Aerosol

EAA Measurements	Diesel	Generator Aerosol
$\bar{D}_{gn}$, μm	0.099[a]	0.14
σ_{gn}	1.88	1.7
$\bar{D}_{gv}$, μm	0.32[a], 0.1[e]	0.33
σ_{gv}	1.88	1.7
Impactor Measurements		
Anderson Impactors (ambient)		
Mass less than 1.1 μm, %		98-99
Diesel Exhaust temp. > 300° C	94-96[b]	
Diesel Exhaust temp. < 300° C	81-94[b]	
Mass less than 0.7 μm, %		91
Mass less than 0.4 μm, %		78
Low Pressure Impactor		
Estimated median particle size, μm		0.08
Transmission Electron Microscopy		
Mean elementary particle size, μm	0.026[b], 0.046[f]	$\bar{D}_{gn} = 0.023\ \sigma_g = 1.4$[c]
Mean agglomerate size, μm		$\bar{D}_{gn} = 0.54\ \sigma_g = 2.2$[d]
Carbon in Aerosol Particles, wt %		
Exhaust temp. > 300° C	98[b]	99
Exhaust temp. < 300° C	89[b]	

[a] Diesel study given in reference [1]

[b] Diesel study from reference [2]

[c] Based on 108 particles from lower two photographs in figure 17

[d] Based on 45 particles from all photographs in figure 16

[e] Reference 14, based on the mean from 5 small diesel engines

[f] Reference 15, based on the mean of 12,000 particle measurements from Catepillar 3150 diesel engine exhaust

TECHNICAL REPORT DATA
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1. REPORT NO.	2.	3. RECIPIENT'S ACCESSION NO.
EPA-600/1-80-014		

4. TITLE AND SUBTITLE	5. REPORT DATE
Carbonaceous Aerosol Generator for Inhalation Studies	February 1980
	6. PERFORMING ORGANIZATION CODE

7. AUTHOR(S)	8. PERFORMING ORGANIZATION REPORT NO.
Thomas G.K. Lee and George W. Mulholland	

9. PERFORMING ORGANIZATION NAME AND ADDRESS	10. PROGRAM ELEMENT NO.
National Bureau of Standards Washington, DC 20234	1AA815
	11. CONTRACT/GRANT NO.
	IAG No. EPA-78-D-X0170

12. SPONSORING AGENCY NAME AND ADDRESS	13. TYPE OF REPORT AND PERIOD COVERED
Environmental Protection Agency Research Triangle Park, NC 27711	Final report 2/78 to 4/
	14. SPONSORING AGENCY CODE
	EPA 600/11

15. SUPPLEMENTARY NOTES

16. ABSTRACT

A carbonaceous aerosol generator designed for inhalation experiments with animals is described. The aerosol produced from a modified diffusion flame has a concentration of 3-10 mg/m^3 at a flow rate of 30 L/min. The addition of a small amount of O_2 to the acetylene fuel greatly increased the efficiency of fuel to particulate conversion, the maximum value was 2.5%. The aerosol size characteristics were: $\overline{D}_{gn}$ = 0.14 µm, based on the electrical aerosol analyzer; $\overline{D}$ ≈ 0.08 µm, based on a low pressure inertial impactor; mediam elementary particle ≈0.023 µm and mediam agglomerate particle ≈0.54 µm, based on transmission electron microscopy. The size characteristics of the generated aerosol are compared with diesel exhaust based on available published data.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Aerosol generator Agglomerate Diesel exhaust Diffusion flame Inhalation Particulates Soot	Carbonaceous Inhalation Toxicology	06F,T

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