

# **SULFATE SAMPLING DILUTION TUNNEL DESIGN AND VALIDATION**

by  
**Melvin N. Ingalls**

**FINAL REPORT  
of  
Task No. 6  
Contract 68-03-2196**

**Prepared for  
Environmental Protection Agency  
Office of Mobile Source Air Pollution Control  
Emission Control Technology Division  
Ann Arbor, Michigan 48105**

**June 1977**



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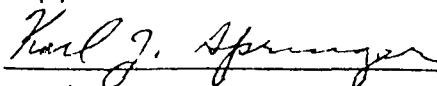
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EPA Project Officer: Richard D. Lawrence

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Ann Arbor, Michigan 48105

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Approved:

A handwritten signature in dark ink, appearing to read "Karl J. Springer", is written over a horizontal line.

Karl J. Springer, Director  
Department of Emissions Research  
Automotive Research Division

## FOREWORD

This project was conducted for the U. S. Environmental Protection Agency by the Department of Emissions Research of Southwest Research Institute (SwRI). Work was begun on April 26, 1976, and completed on June 25, 1977. The project was conducted under Task 6 of Task Order Contract 68-03-2196. It was identified within Southwest Research Institute as Project 11-4291-008.

The EPA Task Officer for this task was Richard D. Lawrence of the Emission Control Technology Division, EPA, Ann Arbor, Michigan. Karl J. Springer, Director, Department of Emissions Research at SwRI served as Project Manager. The task was under the supervision of Melvin N. Ingalls, Senior Research Engineer. The initial tunnel design was done by Terry Ullman, Research Engineer, and the final drawings were made by Bob Nye. Although a number of Department of Emissions Research personnel were involved in the project, key personnel included Nathan Reeh, technician, who constructed the tunnel; Tom Jack, senior technician and Dolores Bynum, laboratory assistant, who assisted in the test phase; and James Herrington, lead technician, and Shelly Stevens, laboratory assistant, who performed the various chemical analyses required.

Several project reviews were held both at SwRI and at the EPA laboratory in Ann Arbor, Michigan. The reviews at SwRI were on April 15, 1976, and November 22, 1976. The reviews at Ann Arbor were on October 1, 1976, and December 10, 1976. In addition to these reviews, frequent telephone conversations provided close liaison between EPA and SwRI during the project.

## ABSTRACT

This report describes the design features and development testing of a dilution tunnel for sampling automotive sulfate emissions. The design of the dilution tunnel and sampling system was the result of a consensus of ideas from representatives of the EPA and industry. The development testing included a propane traverse to verify exhaust mixing; testing the critical flow sampling system; development and use of a sulfate mist generator to determine system sulfate recovery; and evaluation of system losses using automotive exhaust.

The propane traverse demonstrated acceptable exhaust gas mixing. The original design sample flow rate could not be maintained because the pressure drop across the sulfate filter caused the critical flow sample system to unchoke.

While system losses were acceptable using the acid mist generator, the system was found to have unacceptable sulfate losses at tunnel temperatures above 85°C (185°F) when tested with automotive exhaust. These losses appeared to be a function tunnel temperature, with losses increasing as tunnel temperature increased. The losses were minimized by increasing the dilution air flow which lowered the tunnel temperature. Sulfate losses in the connecting tubing between the car exhaust pipe and sulfate tunnel were found to increase considerably as the tubing length increased. At the completion of the development tests, the sampling system, including the tunnel and sampling and control console, was delivered to EPA, Ann Arbor. A complete set of drawings of the tunnel was furnished with the tunnel.

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## SUMMARY

A dilution tunnel and sulfate sampling system to be used with both the positive displacement pump (PDP) and critical flow venturi (CFV) constant volume sampler (CVS) emission sampling systems was built from EPA design criteria. The system uses a 2.7 m (8.8 ft) long by 21 cm (8.4 in.) inside diameter stainless steel pipe as the dilution tunnel. Exhaust mixing length in the tunnel is approximately 1.74 metres (68.5 in.).

The sulfate probe is of 2.54 cm (1 in.) stainless steel tubing approximately 61 cm (24 in.) long with a removable tip which reduces the probe diameter to that necessary to provide isokinetic sampling. For the CFV-CVS system, this removable tip contains a convergent-divergent section designed to operate in the choked flow regime. The probe inlet is centered in the tunnel, with the holder for the sulfate filter located outside the tunnel,

An alarm system is furnished to provide an audible and visible alarm whenever the tunnel temperature is outside a specified range. Another alarm is activated whenever the pressure downstream of the probe critical flow nozzle increases to the point that the nozzle flow becomes unchoked. Controls are also provided for the air heaters installed in the dilution air plenum.

The development testing performed included a propane traverse to determine the extent of exhaust mixing; proof testing of the CFV-CVS sampling system; development and use of a sulfate mist generator to determine system sulfate recovery; and evaluation of system losses using actual automotive exhaust. The propane traverse showed that the maximum and minimum propane concentrations were within approximately  $\pm 5$  percent of the average. This spread was considered small enough not to affect the accuracy of the sulfate sample.

A SET-7\* test was run with the critical flow nozzle sample probe used with CFV-CVS units. The test revealed that the originally planned sample rate of  $0.028 \text{ m}^3/\text{min}$  (1 cfm) resulted in the nozzle unchoking as the pressure drop across the filter increased due to sulfate loading. As a result, the sample flow was changed to  $0.014 \text{ m}^3/\text{min}$  (0.5 cfm) for both the PDP and CFV-CVS systems.

A sulfuric acid mist generator was developed which injected sulfuric acid into the tunnel at a rate approximating automotive exhaust. Approximately 80 percent of the mist generated was less than one micron in size. The sample filter recovered an average of 105 percent of the acid injected. Accuracy limits of the analytical procedures are probably responsible for recoveries greater than 100 percent. Improved analytical methods were developed for future work. Probe losses were approximately 3 percent at a tunnel temperature of  $55^\circ\text{C}$  ( $131^\circ\text{F}$ ). While a great deal of progress was made on both the mist generation and sulfate recovery procedures, more work is needed to perfect both.

Tests with car exhaust revealed that probe and tunnel losses were a function of tunnel temperature, increasing as temperature increased. The total system losses (excluding the connecting piping) varied from 3.9 percent at  $68^\circ\text{C}$  ( $155^\circ\text{F}$ ) to 21.6 percent at  $96^\circ\text{C}$  ( $205^\circ\text{F}$ ). The type of probe material appeared to make some difference in the amount of sulfate lost on the probe. However, at  $85^\circ\text{C}$  ( $185^\circ\text{F}$ ) average temperature probe losses were above 25 percent with stainless steel, Teflon and glass probes. There was also some indi-

\* SET-7 is the congested freeway driving schedule.

cation that the system would become "seasoned" and that sulfate losses would decrease with test time. A test of the critical flow sample probe nozzle showed higher sulfate losses than the noncritical flow nozzle (15% vs. <3%) at about 70°C tunnel temperature.

For this project, a 1588 kg (3500 lb) inertia weight car with a 4.98 litre (304 CID) engine was used. The tunnel temperature during SET-7 tests run with this car were higher than permissible for acceptable sulfate losses. A brief check of exhaust temperatures of several cars revealed that for many cars, a 9.20 m<sup>3</sup>/min. (325 cfm) CVS may not produce low enough tunnel temperatures for acceptable sulfate losses. To minimize system sulfate losses the tunnel temperature should be as low as possible, yet still above the dew-point of the dilute exhaust. To do this, either the dilution air temperature must be lowered or its volume increased. The choice for this study was to increase the volume.

A 14.73 m<sup>3</sup>/min (520 cfm) CVS was used for a series of tests with the same test car used in the previous tests. For a SET-7 test, the average temperature was reduced to 59°C (138°F) from about 85°C (185°F) for a SET-7 test with a 9.20 m<sup>3</sup>/min (325 cfm) CVS.

During these tests the sample probe with the critical flow nozzle was used. Probe losses with the critical flow probe averaged 19 percent compared with the less than 3 percent for the noncritical flow probe at a CVS flow of 9.2 m<sup>3</sup>/min (325 cfm) and approximately the same average tunnel temperature. While time did not permit further investigation of the sulfate losses with the critical flow probe, it is recommended that a thorough investigation of the losses with this probe be conducted prior to using this system for routine sulfate testing.

Two different lengths of connecting tubing between the car exhaust and the tunnel were tested for sulfate losses. The car was run for 25 minutes at 64 km (40 mph) to generate the exhaust sulfates. The loss in the 0.61 metre (2 foot) pipe was approximately 0.6 percent; the loss in the 4.57 metre (15 foot) pipe was approximately 20 percent.

The sampling system, including pump, flow meters and controls was fitted into a console together with the alarm system and heater controls. This console and the dilution tunnel itself were shipped to EPA, Ann Arbor at the completion of the test phase of this project. A complete set of drawings of the tunnel was also furnished with the system.

## CONCLUSIONS

The following conclusions can be drawn from the testing performed with the 21 cm diameter dilution tunnel.

1. Tunnel and sample probe sulfate losses are a function of tunnel temperature, with losses increasing as temperature increases.
2. The sulfuric acid mist generator developed during this project can be used to determine system sulfate recovery, however the procedure needs further refinement.
3. The tunnel design provides adequate mixing of car exhaust gas and dilution air.
4. There is considerable sulfate lost when connecting tubing on the order of five metres long is used between the car exhaust and the dilution tunnel.
5. The sample flow rate for the critical flow sample probe nozzle designed for use with a CFV-CVS should be at or below  $0.014 \text{ m}^3/\text{min}$  (0.5 cfm) because of high filter pressure losses.
6. Sulfate losses using the critical flow sample probe nozzle are higher than losses on the noncritical flow nozzle at the same tunnel temperature.
7. For many cars, a  $9.20 \text{ m}^3/\text{min}$  (325 cfm) capacity CVS may not produce low enough tunnel temperatures for acceptable sulfate losses without precooling the dilution air.

## I. INTRODUCTION

Past EPA studies showed that cars equipped with catalytic converters emitted more sulfates than noncatalyst cars.<sup>(1-3)\*</sup> With the possibility of the routine testing of prototype cars for sulfate emissions, the need for a standardized sulfate sampling system became evident.

In an effort to develop an acceptable standard sulfate sampling system for use by the EPA Mobile Source Pollution Laboratory at Ann Arbor, Michigan, and hopefully by the auto industry as well, a meeting was held on December 4, 1975 at the EPA, Ann Arbor, facility with representatives of the EPA, the auto manufacturers, and Southwest Research Institute in attendance. From this meeting a general design consensus and criteria for a sulfate sampling system emerged.

### A. Objective

The objective of this project was to build and test a suitable sulfate dilution tunnel and filter collection system based on SwRI's mini-tunnel design which was used extensively in previous sulfate testing projects for EPA (2,4). The new tunnel was to use design criteria established at the December 4, 1975 meeting at Ann Arbor between EPA and industry. Once built, the tunnel was to be used to investigate ways to achieve a calibration with sulfuric acid mist; determine the effect of exhaust connecting tubing length; determine the effect of dilution air humidity; and compare this 21 cm (8 inch) diameter tunnel to a 46 cm (18 inch) diameter tunnel.

### B. Approach

To meet the objectives of the project, a 12 item Statement of Work was agreed upon by EPA and SwRI. The complete Statement of Work is included as Appendix A. The Statement of Work was later modified to include furnishing the necessary hardware for a complete sampling system and to add additional testing to better define the system sulfate losses discovered during the test program.

Briefly the plan was to: (1) build the tunnel to the agreed upon specifications; (2) develop a sulfuric acid mist injection system; (3) determine the sulfate sampling system recovery using the sulfuric acid mist injection system; (4) compare the sulfate levels obtained from steady-state and sulfate cycle tests on an actual car with sulfate levels obtained using an 18 inch diameter tunnel on the same car and test cycles and; (5) evaluate the effects of connecting tubing length between car and tunnel using a 2 foot long piece of flexible tubing. At the completion of the test program, a complete set of drawings would be made incorporating any changes found necessary during the test program.

During the project the test plan was modified to delete the comparison of the small tunnel and the 18 inch diameter tunnel and to delete the tests

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\*Superscript numbers in parentheses refer to the List of References at the end of this report.

to determine the effect of humidity. In place of these tests, a series of tests were performed to define the system losses as a function of tunnel temperature.

## II. DESIGN FEATURES

The design considerations and physical layout of the sulfate tunnel, probe and sampling and control system are explained in this section of the report.

### A. Preliminary Design Considerations

As mentioned in the Introduction, a planning meeting was held in Ann Arbor between EPA and industry for the purpose of agreeing on a set of design parameters for a sulfate dilution tunnel sampling system. The following parameters were agreed upon:

The dilution tunnel diameter will be approximately 8 inches

The distance between the exhaust inlet and the probe tip will be 8 pipe diameters

The distance between the probe tip and the downstream end of the tunnel will be 2.5 pipe diameters

The tunnel material will be stainless steel

A mixing orifice will be located at the plane of the exhaust inlet

The probe will be made of stainless steel tubing

The filter holder will be outside the tunnel

The EPA added the following requirements to meet their particular needs:

Provide mixing of the exhaust and dilution air

Be compatible with a CVS flow rate of 325 SCFM at typical dilution tunnel operating conditions of 735 mm HgA and 66°C

Be compatible with EPA certification test cells

Sample system to be able to be used with a critical flow venturi (CFV)-CVS and a positive displacement pump (PDP) - CVS

### B. Tunnel Design

From the criteria listed above the basic tunnel system was chosen. The tunnel was constructed of 8 inch schedule 5 welded stainless steel pipe. This pipe had an inside diameter of 21.4 cm (8.41 inches) and a wall thickness of 2.77 mm (0.109 inches). This fixed the distance between the exhaust inlet plane and the probe tip at 170.7 cm (67.2 inches), and the distance between the probe tip and the tunnel exit at 53.3 cm (21.0 inches). The

overall length of the tunnel is 2.67 metres (8.75 feet). Provision was made for the exhaust to enter from either side of the tunnel using a 7.6 cm (3 inch) diameter elbow through a 15.2 cm (6 inch) Tri-Clover (Ladish Co.) coupling. The exhaust discharges into the tunnel in the downstream direction. A 15.2 cm (6 inch) mixing orifice is located in the tunnel at the plane of the exhaust discharge. The diluted exhaust exits the tunnel through a nominal 4 inch stainless steel pipe approximately 11 cm (4.5 inches) long. A 15.2 cm (6 inch) Tri-Clover coupling installed in the top rear of the tunnel is used for the sample probe port. To better fit the EPA certification test cells and utilize minimum floor space, the dilution air filter box was placed above the tunnel. In addition to the usual filters, the dilution air inlet plenum was equipped with duct heaters to allow the sulfate tunnel to be pre-heated. It also allows the tunnel to maintain a minimum temperature above the condensation temperature of the mixed stream during a test. Figure 1 is a schematic of the tunnel showing some of the more pertinent dimensions. Figure 2 contains photographs of the tunnel from various angles.

### C. Sample Probe Design

The sample probe was made from 2.54 cm (1 inch) stainless steel tubing with 1.24 mm (0.049 inch) wall thickness and is approximately 61 cm (24 inches) long. The probe is fitted with an interchangeable nozzle tip(s) to provide isokinetic sampling. When installed, the probe tip points directly upstream. There is a single 45 degree bend in the probe. The probe comes out of the tunnel at a 45 degree angle through a 15.2 cm (6 inch) Tri-Clover coupling. The filter holder is located outside the tunnel at the end of the probe. The initial sample flow rate specified by EPA was 0.028 m<sup>3</sup>/min (1 cfm).

Part of the design criteria for the tunnel was that the sulfate sampling system be able to be used with a CFV-CVS. The CFV-CVS is in reality a proportional sampler. Since the flow is controlled by a critical flow venturi, the volumetric flow is a function of the gas stream pressure and temperature. The gaseous emissions sample lines of a CFV-CVS also have choked flow sections so that the sample taken is proportional at all times to the total volume flowing through the CFV-CVS. Therefore, the sulfate sampling system must also be proportional at all times to the total volume flowing through the CVS. To do this, the sulfate sampling system must also have a critical flow section. After much discussion of the best way to accomplish this, it was decided to use a choked convergent-divergent nozzle at the sample probe inlet. This arrangement will give sulfate sample flow proportional to the total CFV-CVS volume flow, providing the temperature difference between the sulfate probe entrance and the CVS venturi is negligible.

Using a total pressure traverse previously performed on a similar tunnel, the velocity at the center of the tunnel was found to be 125 percent of the bulk velocity. See Appendix B. Thus, for a 325 cfm CVS at 735 mm of mercury and 66°C, the velocity seen by the probe is 5.89 m/sec (19.3 ft/sec). This fixed the nozzle inlet diameter for 0.028 m<sup>3</sup>/min (1 cfm) at 10.1 mm. Using standard compressible flow calculations<sup>(5)</sup>, the nozzle throat was calculated to be 1.59 mm.

For smooth transition, the angles for the converging and diverging section

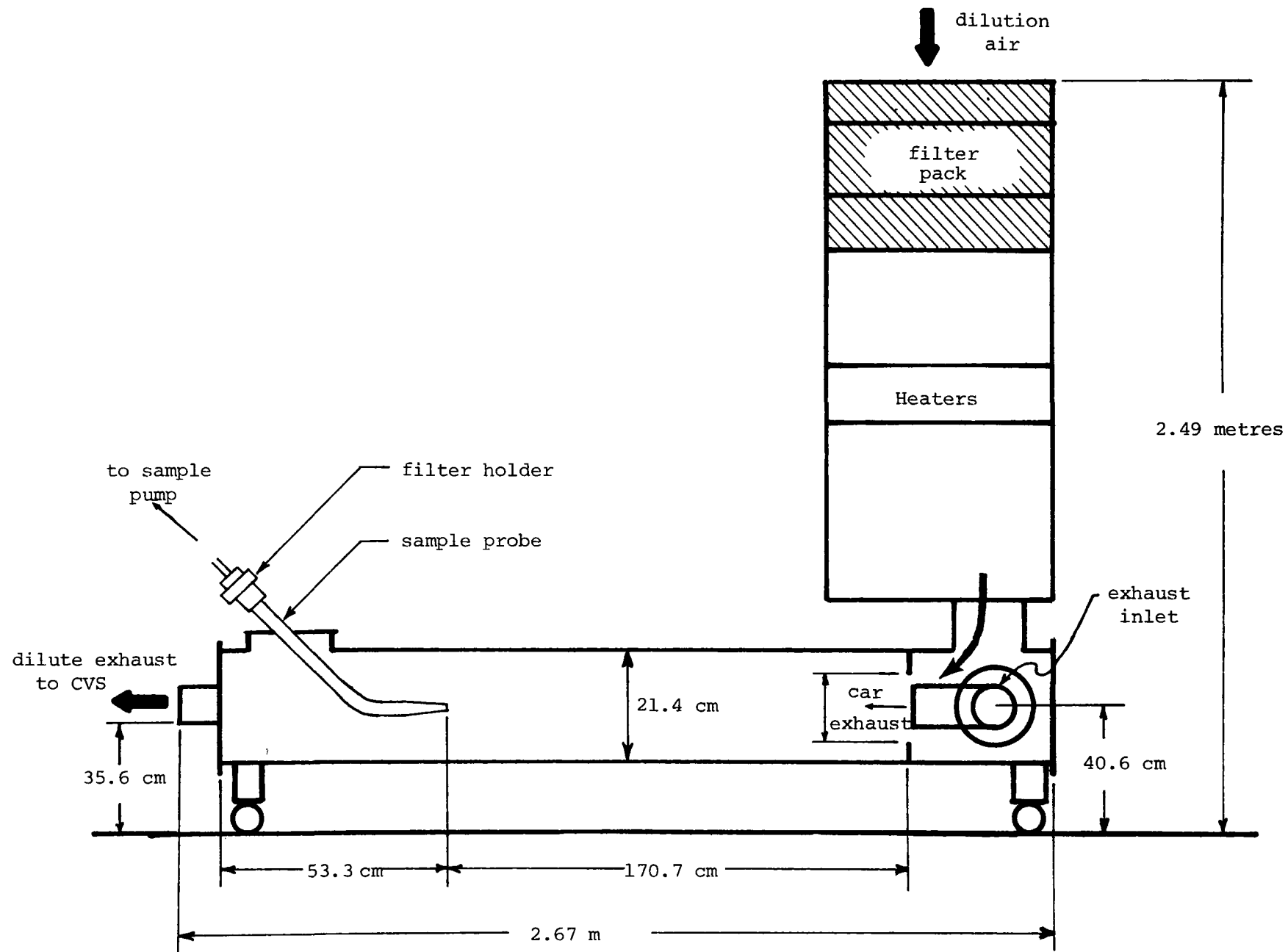


Figure 1. Schematic of Sulfate Sample Dilution Tunnel



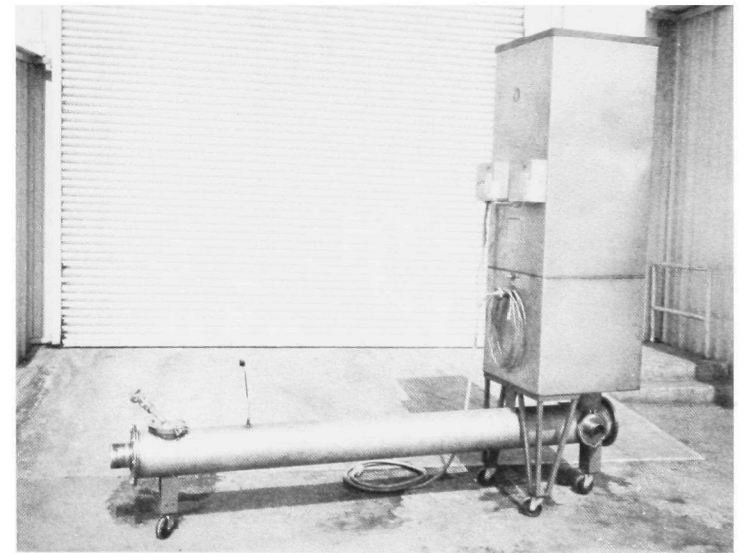
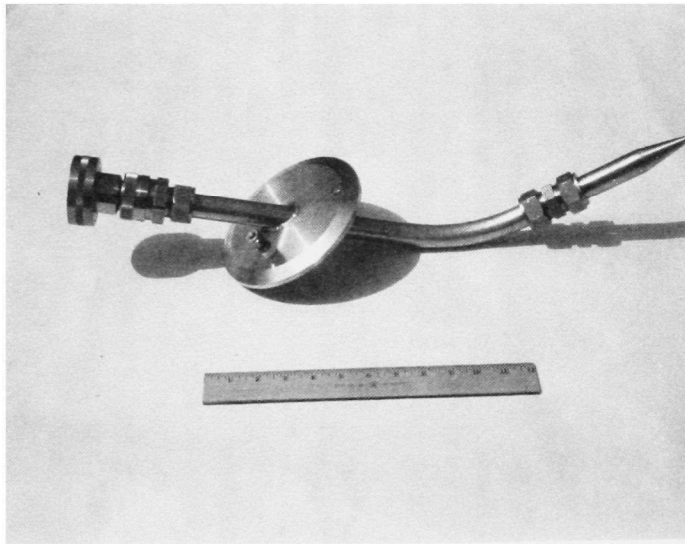
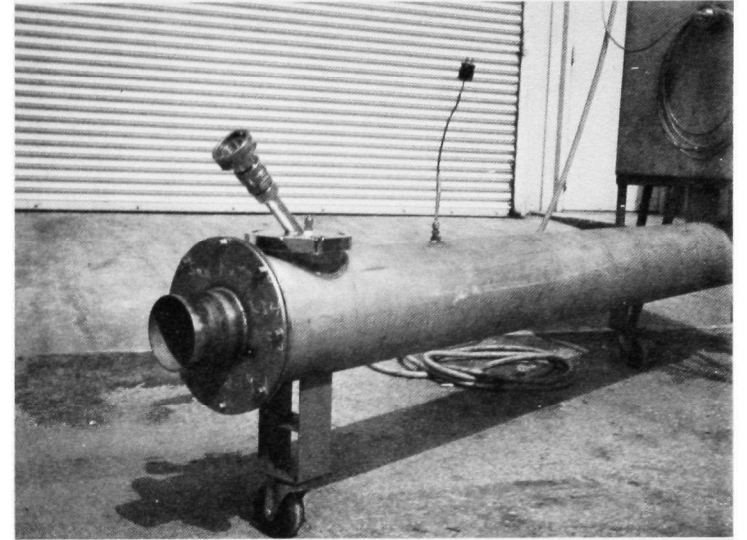
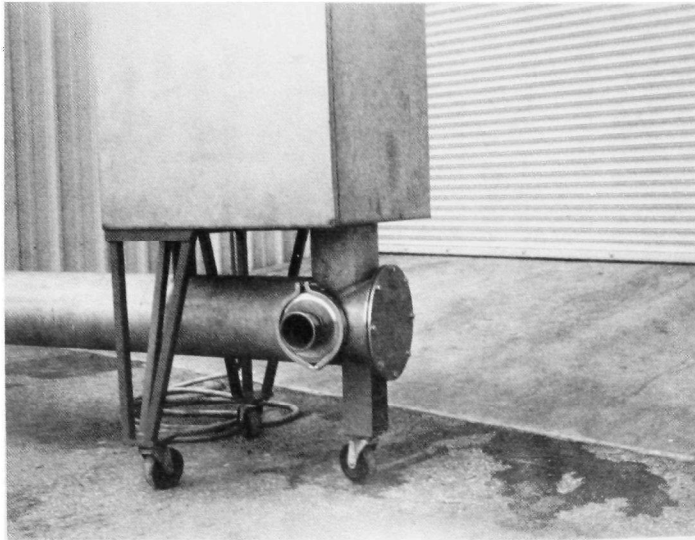


Figure 2. Photographs of Sulfate Dilution Tunnel

of the nozzle were set at 15 degrees for each. The nozzle length was set so that the entrance was tapered for approximately 2 nozzle diameters.

For a positive displacement pump (PDP) CVS the critical flow section is not used. Since the testing to be performed at SwRI would be with a PDP-CVS, a second nozzle was designed without the converging-diverging section. Sketches of both nozzles are shown in Figure 3.

At the request of the EPA, the standard 47 millimeter Fluoropore (Millipore Corporation) filterholder was modified with quick connects on each end. The inlet end was replaced with a new, longer, diverging section, and a Viton "O" ring to seal the filter. The male part of a 1 inch quick connect was machined into the end of the filter holder entrance piece. The discharge side was drilled and tapped with 1/2 inch pipe threads and the stem of a 1/2 inch full flow quick connect installed. A sketch of the filter holder is shown in Figure 4.

#### D. Sampling and Control System

The original intent of this project was that SwRI would provide to EPA, Ann Arbor, only the sulfate tunnel and probe, with the EPA to furnish the remainder of the sampling system. During the course of work, the EPA modified the scope of the project to include a complete sampling and control system. A picture of the sampling and control console is shown in Figure 5. The console contains: (1) sample system including pump, flow meters and controls; (2) alarm system to indicate loss of critical flow and failure to remain within temperature limits; and (3) dilution air heater controls.

The sample system consists of a sample drier tube filled with a molecular sieve, a Gast Model 2565 vacuum pump, a rotameter for visual indication of instantaneous flow rate and an American Meter Division Gas Sampling meter for total sample volume measurement. An interconnect is provided to allow the sample pump to be turned on and off from the CVS control panel.

The alarm system was designed at the request of the EPA to provide an audible and visible alarm whenever the tunnel temperature is outside a specified temperature range (high or low). Another alarm is activated whenever the pressure behind the sulfate sample probe critical flow nozzle increases to the point that the nozzle becomes choked.

The controls for the heaters in the dilution air plenum allow the amount of heating to be controlled manually or automatically. Three different levels of heating are provided under manual operation. A temperature controller provides for automatic heater control for a given tunnel temperature. It should be recognized that the temperature controller provides control on the minimum tunnel temperature only. During a test, if the dilute exhaust temperature exceeds the tunnel set point temperature, the heaters would shutoff and the tunnel temperature would be dependent on the car exhaust and ambient air temperatures until the tunnel temperature again fell below the set point. An interconnect is also provided to permit the heaters to be operated only when the CVS blower is on. This keeps the heater elements from burning-out from overheating.

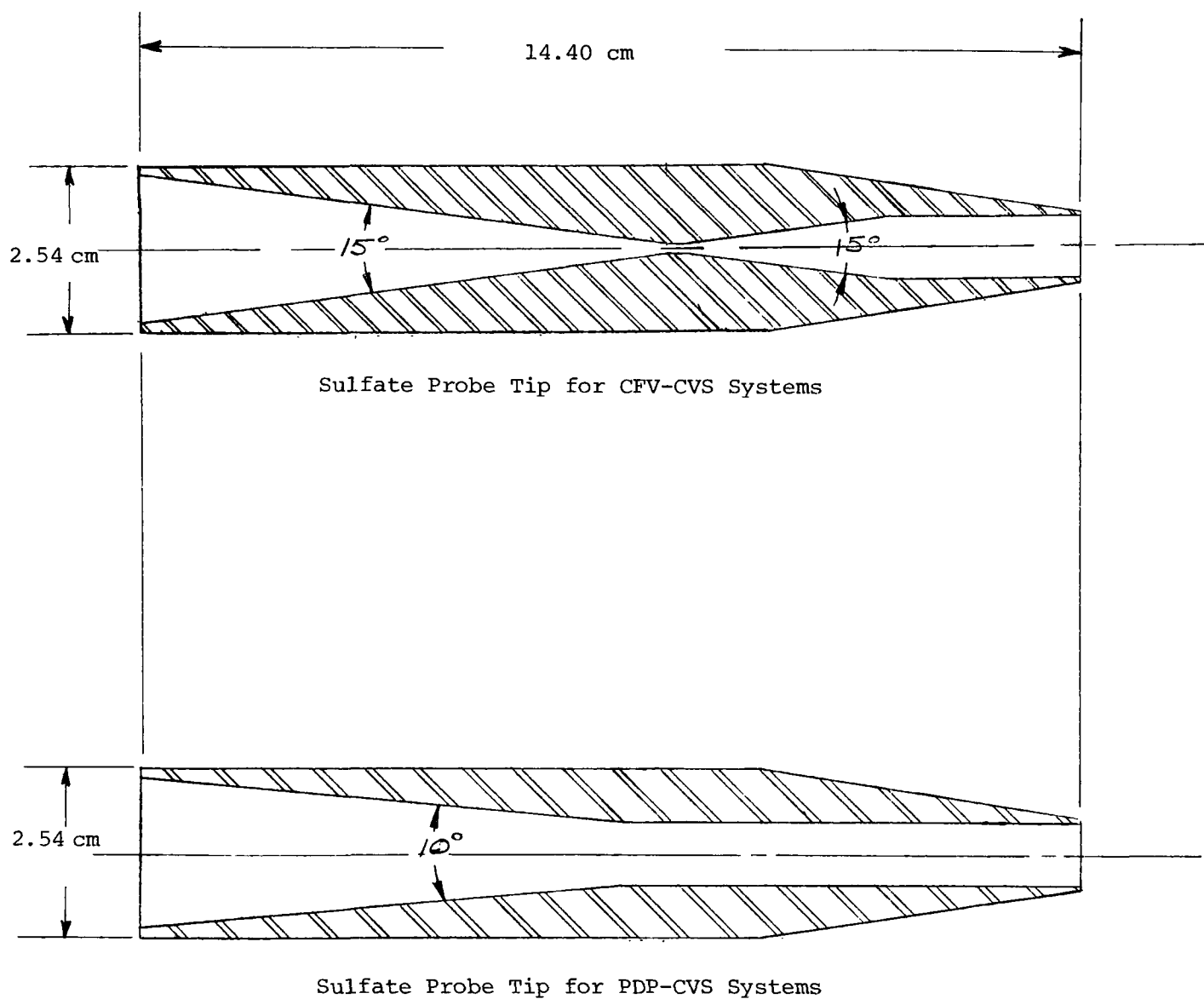
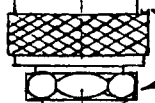
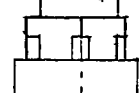
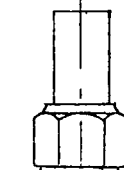
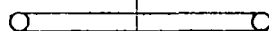
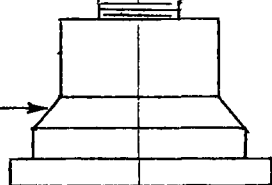


Figure 3. Sulfate Sampling Probe Tips

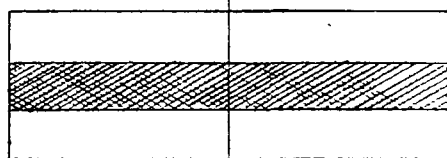
To Sample Pump



Modified Discharge side  
of Millipore 47 mm filter holder

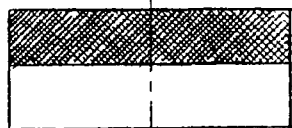
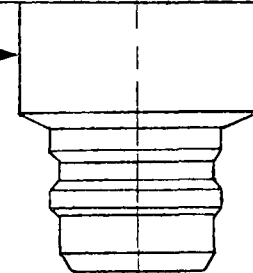


Viton "O" Ring

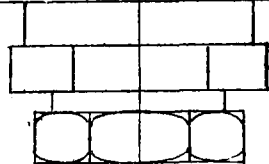


Closure ring for  
Millipore 47 mm  
Filter Holder

Redesigned inlet portion of  
filter holder with integral  
quick connect coupling stem

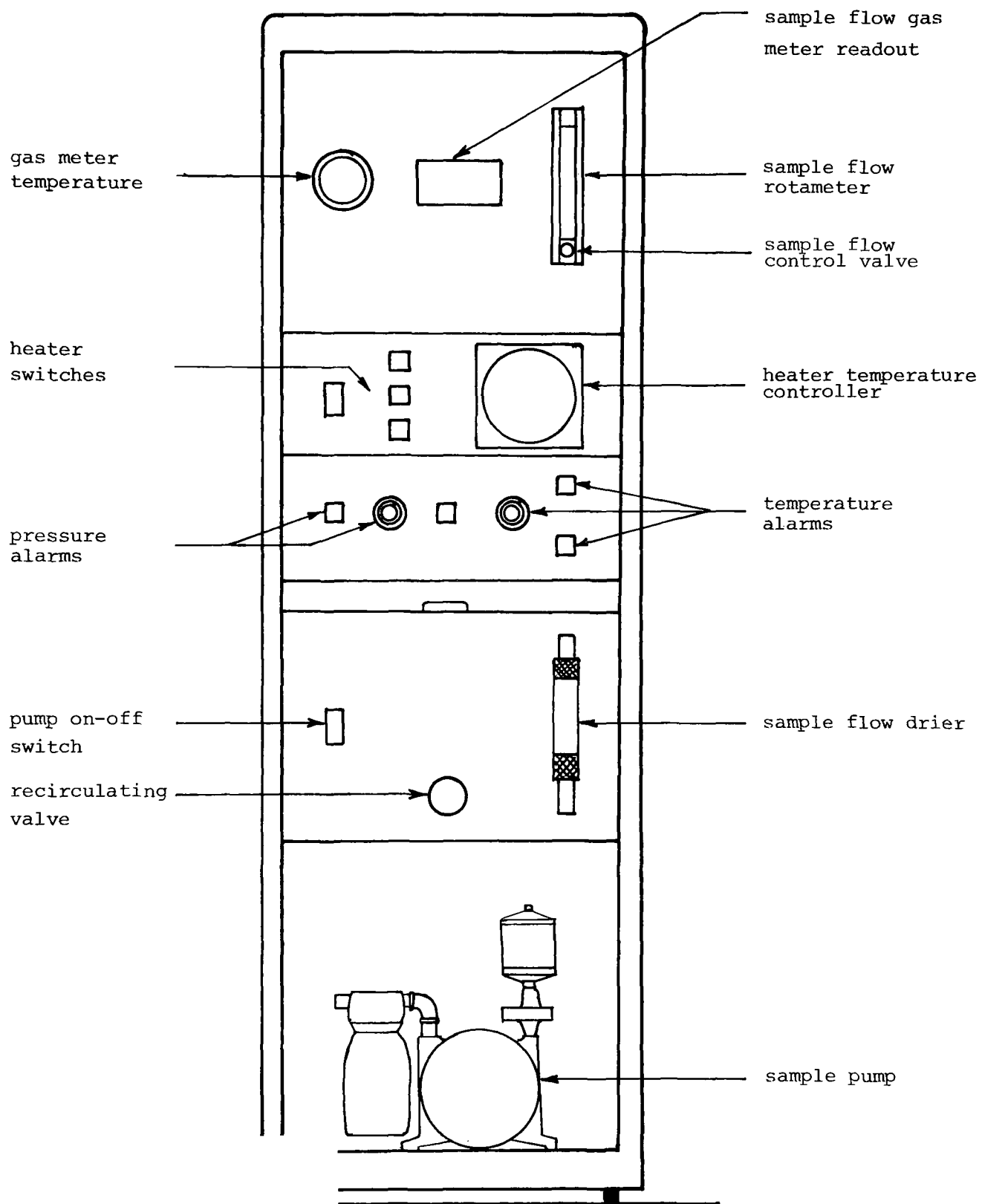


Swagelok 1" full flow quick  
connect coupling body



From Sample Probe

Figure 4. Modified 47 mm Filter Holder



Sampling and Control Console

#### E. Design Modifications Resulting From Test Program

As the result of the tests described in Sections III, IV and V, some modifications to the sampling system were required. The CVS volume flow was increased to 15 m<sup>3</sup>/min (530 cfm) with 19.8 m<sup>3</sup>/min (700 cfm) as an alternative for the CFV-CVS and the sulfate sample flow decreased to 0.014 m<sup>3</sup>/min (0.5 ft<sup>3</sup>/min). This required the resizing of the sulfate sample probe tips. While the internal dimensions changed somewhat, the configuration of each tip remained the same as that shown in Figure 3.

### III. SULFATE MIST GENERATOR EXPERIMENTS

One of the goals of this project was to do preliminary development and evaluation of a sulfate injection method of calibrating the complete tunnel sampling system similar to the propane test used for the CVS gaseous emission sampling system. This section of the report covers the development of the mist generator and its use in determining system recovery.

#### A. Development of Mist Generator

The first step in developing a sulfate calibration system was finding a mist generator that would produce the required mist size (98 percent less than 1 micron) in an amount equivalent to an automobile exhaust (about 7 mg/min of  $\text{H}_2\text{SO}_4$ ). The design of the system should also be such that it is relatively easy to determine the amount of sulfuric acid injected into the tunnel. Several mist generators were investigated including Collison type generators from several manufacturers, Sierra Instruments Spinning Disc Aerosol Generator, the "Misto-Gen" ultrasonic generator, and others. For most of these generators, it appeared to be difficult to determine accurately how much sulfuric acid mist would be generated for a given test.

After discussing the problem with Dr. Paul Lamoth of the EPA (Research Triangle Park, North Carolina), who was working in the field of atmospheric monitoring of aerosols and had used several of the mist generators under consideration, it was decided to use the calibration mist generator from a Baird Atomic atmospheric sulfuric acid analyzer.

This mist generator was all glass with short flow passages, so that it would be easy to recover the residual sulfuric acid from the generator. A drawing of this generator is shown in Figure 6.

The generator produces a mist by breaking up droplets of dilute sulfuric acid with compressed air. The acid is admitted into the generator through a capillary tube attached to a small funnel. As the acid drips into the mist chamber, it hits the air jet from the compressed air nozzle and is broken up into a mist. A piece of glass tubing connects the generator to the tunnel.

Not all of the acid entering the chamber left as an aerosol mist. The larger particles from the droplet impinged on the side of the chamber and ran down to the bottom of the chamber. There was also considerable condensation on the walls of the mist generator.

The original generator was modified by placing a stopcock in the chamber drain line. If the chamber became too full, some of the residual acid was drained from it. However, some liquid was always left in the chamber so that no mist would escape through the drain.

The proposed test procedure began with feeding a given amount of dilute sulfuric acid of known normality into the mist generator. Knowing the amount and normality of the dilute solution, the amount of sulfate put into the generator could be calculated. After the test, the residual acid would be drained from the generator and the generator and discharge tube rinsed with a small amount of distilled water. The rinsing would be added to the residual acid and the total volume measured. The normality of the mixture would be determined by titrating an aliquot of the mixture with sodium hydroxide. From the

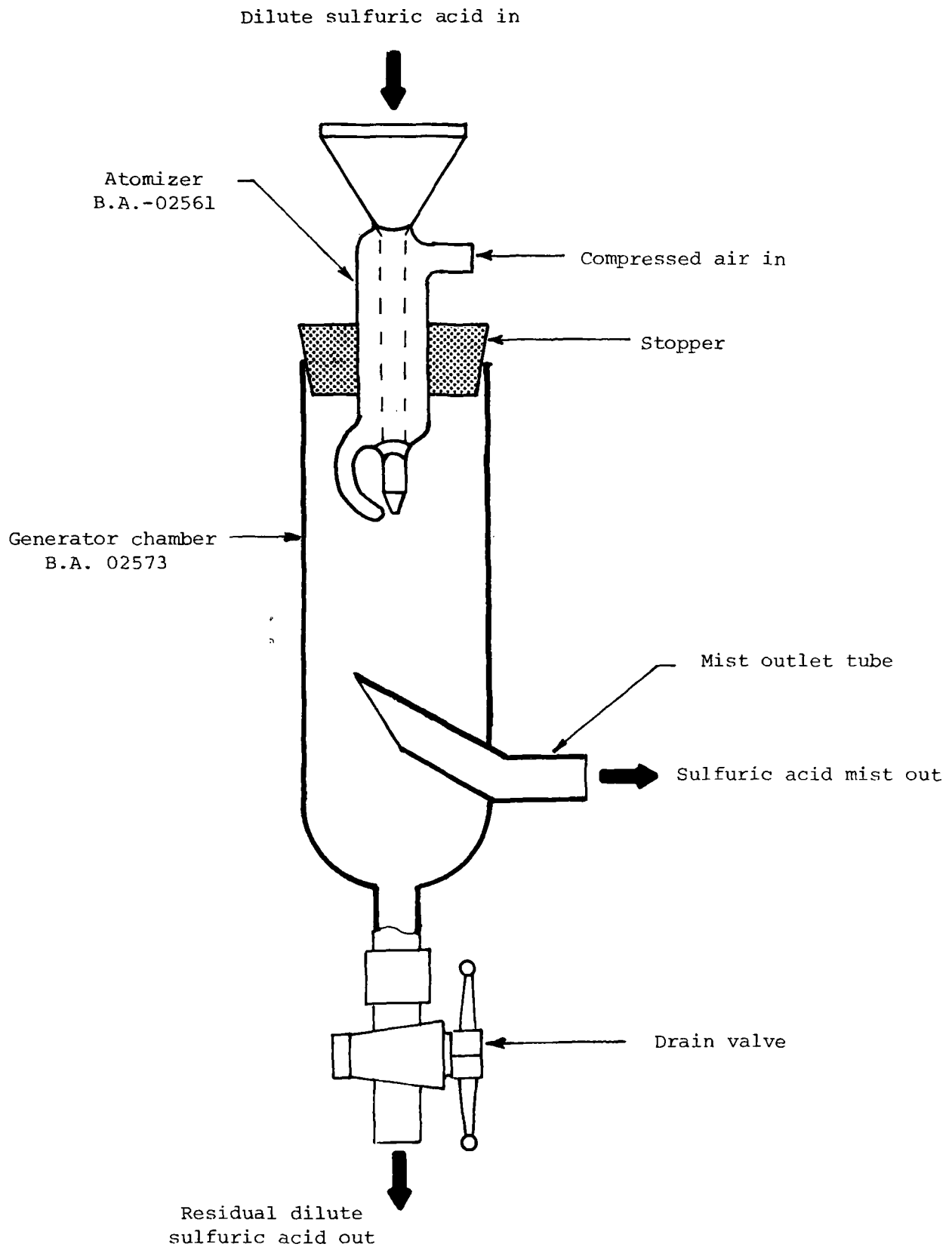


Figure 6. Baird-Atomic Mist Generator



volume and normality of the mixture, the amount of sulfate remaining in the generator could be determined. Subtracting this amount from the total sulfate put into the generator would give the amount of sulfate injected into the tunnel.

Preliminary bench tests were run with the generator using water to determine the amount of liquid converted to mist, and to determine the air pressure that gave the highest mist generation rate. The data from these tests is contained in Appendix C. It was found that approximately 12 percent of the dilute acid feed into the generator was atomized into mist. The optimum air pressure was found to be about 138 kilopascals (20 psi). From these tests, it was decided to use 100 ml of 0.2 N sulfuric acid as the mist generator feedstock. Figure 7 shows the mist generator set up to inject mist into the tunnel.

#### B. Aerosol Sizing Tests

An Andersen impactor was chosen as the easiest to use and most readily available device for determining the size distribution of the sulfate aerosol in the tunnel. Figure 8 shows the details of the impactor. The Andersen Stack Sampling Head consists of a stainless steel case and 1/2" female pipe fittings at the inlet and discharge. It is designed to be inserted directly into the stack.

The Sampler contains nine jet plates each having a pattern of precision-drilled orifices. The nine plates, separated by Teflon "O" rings, divide the sample into eight fractions or particle size ranges. The jets on each plate are arranged in concentric circles which are offset on each succeeding plate. The size of the orifices is the same on a given plate, but is smaller for each succeeding downstream plate. Therefore, as the sample is drawn through the sampler at a constant flow rate, the jets of air flowing through any particular plate direct the particulates toward the collection area on the downstream plate directly below the circles of jets on the plate above. Since the jet diameters decrease from plate to plate, the velocities increase such that whenever the velocity imparted to a particle is sufficiently great, its inertia will overcome the aerodynamic drag of the turning airstream and the particle will be impacted on the collection surface. Otherwise, the particle remains in the airstream and proceeds to the next plate. Since the particle deposit areas are directly below the jets, seven of the plates act as both a jet stage and a collection plate. Thus, No. 0 plate is only a jet stage and No. 8 plate is only a collection plate.

For this project, a backup 0.5 micron Fluoropore filter was placed on a plate 8 to trap all the remaining particulate. It should be pointed out that the impactor is calibrated in terms of unit density (1 g/cc). It can be readily seen that two objects of the same size, but different densities, will have different inertial characteristics and would behave differently in different environments. By referencing the sizes to unit density, the particles are measured in what is termed aerodynamic size. Particles of the same aerodynamic size behave aerodynamically as though they were the same size, even though they have different physical sizes and densities.

The size distribution was determined by washing each of the nine impactor plates with 20 ml of 60 percent isopropyl alcohol solution and processing the solution using the Barium Chloranilate (BCA) procedure employed with sulfate filters.<sup>(6)</sup> The backup filter was processed using the usual BCA procedure for sulfate filters.

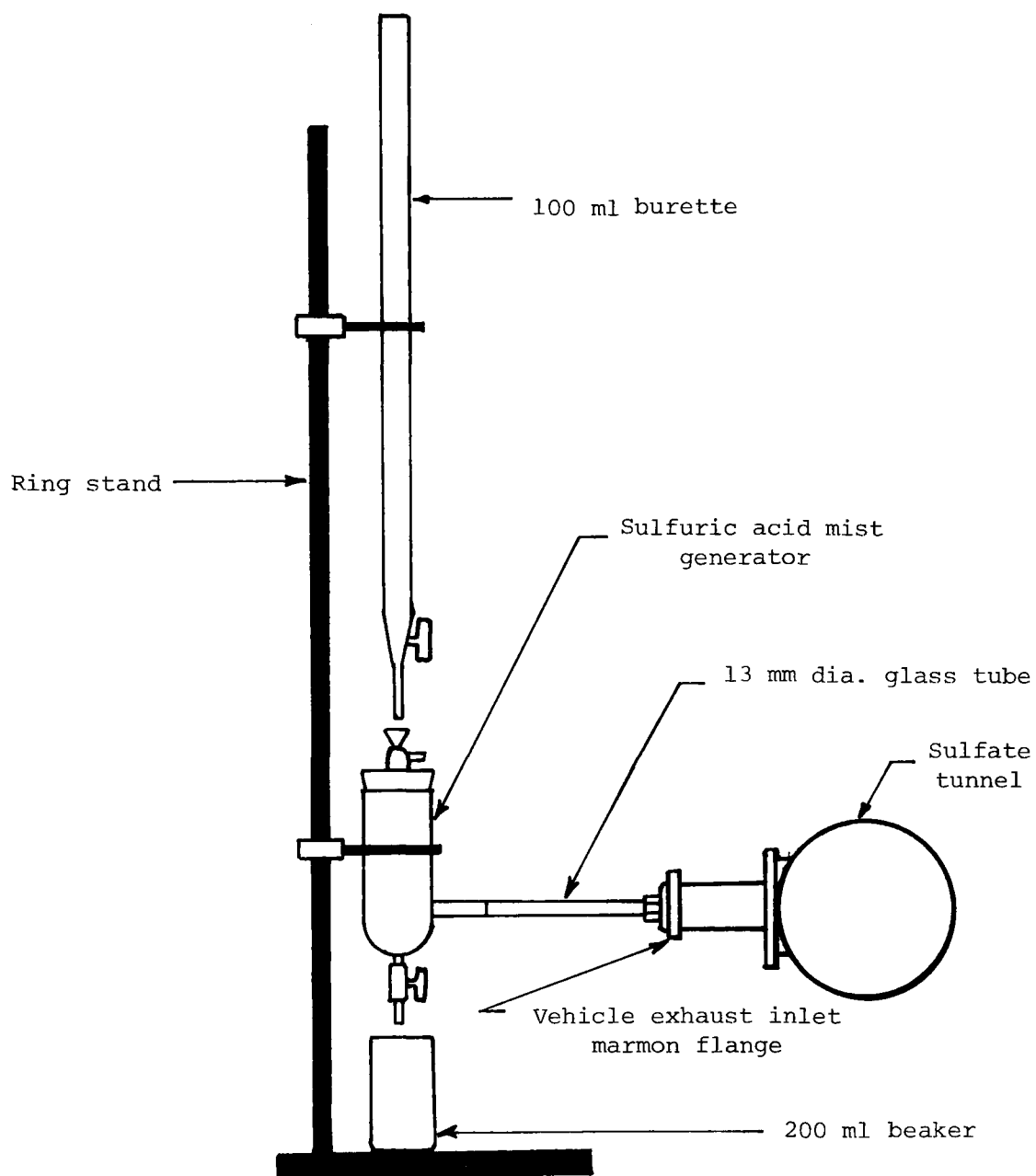


Figure 7. Sulfate Mist Injection Equipment

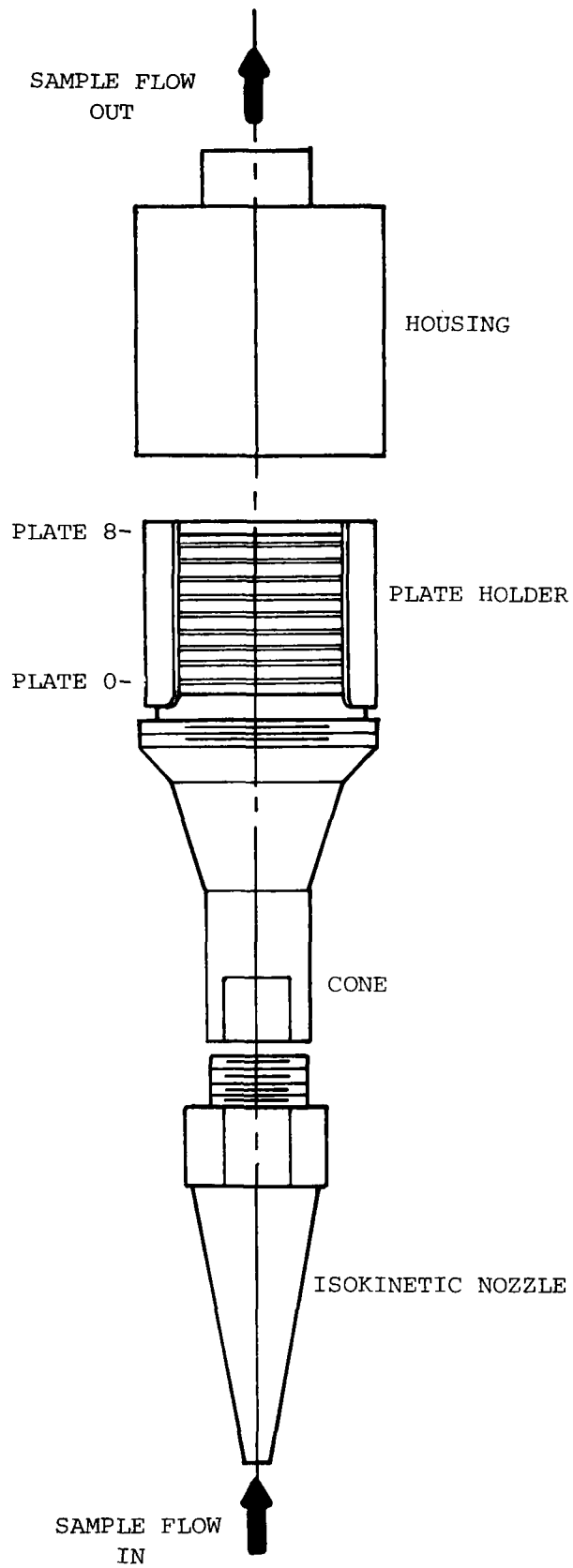


Figure 8. Andersen Impactor

The test set-up for the sizing tests using the Andersen impactor is shown in Figure 9. The impactor is suspended in the tunnel at the same location that is normally occupied by the sulfate probe. Preliminary tests showed that while the Baird Atomic mist generator produced a sufficiently fine mist, it did not produce a sufficient quantity for the desired accuracy of size determination from the impactor plates. Since the size distribution was determined from the amount of sulfate on the plates, the more sulfate that could be put through the system, the more accurately the percent of each size could be determined. Also, it was desired to have the amount of sulfate be close to that generated by car exhaust. The target level was about 130 mg per test.

In an effort to increase the quantity of mist, the generator chamber was lightly heated by wrapping it with electrical heating tape. While this increased the amount of aerosol to between 30 and 40 mg per test, this was not deemed sufficient to represent automotive exhaust. The mist generator was then modified by increasing the size of the chamber as shown in Figure 10. The objective of this modification was to allow more room in the generator chamber, reducing losses due to impingement of the droplets on the walls. The modified generator did produce a greater amount of aerosol. Although the amount of aerosol was still not quite to the level desired, it was deemed adequate for development tests and the configuration was used for the remainder of the project.

A total of nine sizing experiments were run, six of which were usable--two tests with the original generator and four with the modified generator. Each test consisted of placing the impactor in the tunnel and drawing a 22.7 l/min (0.8 cfm) sample through the impactor while sulfuric acid mist was being injected into the tunnel. The mist generator was fed with 100 ml of 0.2 N sulfuric acid. This normally required about 25 to 30 minutes. The total sulfuric acid injected into the tunnel varied from approximately 35 mg for the original generator to approximately 60 to 110 mg for the modified generator.

The results of the sizing tests are shown in Figure 11. The amount of sulfate mist below 0.4 microns (except for one test) varied from approximately 50 to 72 percent. The highest value was obtained with the sulfate mist generator in its original configuration. The lowest percentage was obtained with the revised mist generator configuration. One test (10/22/76) showed only 25 percent of the mist below 0.4 micron. The reasons for this are not immediately apparent. It may have to do with the amount of heating of the generator, or it may be the result of high residual sulfates on some of the sampler plates. It was extremely hard to remove all residual sulfate from the plates. Sometimes the residual sulfate indication would increase between two successive cleanings.

Several things were learned from this series of tests. The first is that the Andersen Impactor, though easy to use, has several deficiencies for this type of aerosol sizing. It cannot separate particles smaller than 0.36 micron at the flow rate used. At the outset of this project, available information indicated that an aerosol mist below 1 micron would be sufficient for simulating automotive exhaust. However, during the project, results from the General Motors sulfate dispersion experiment<sup>(7)</sup> became available which indicated that exhaust sulfate particle size in the atmosphere was less

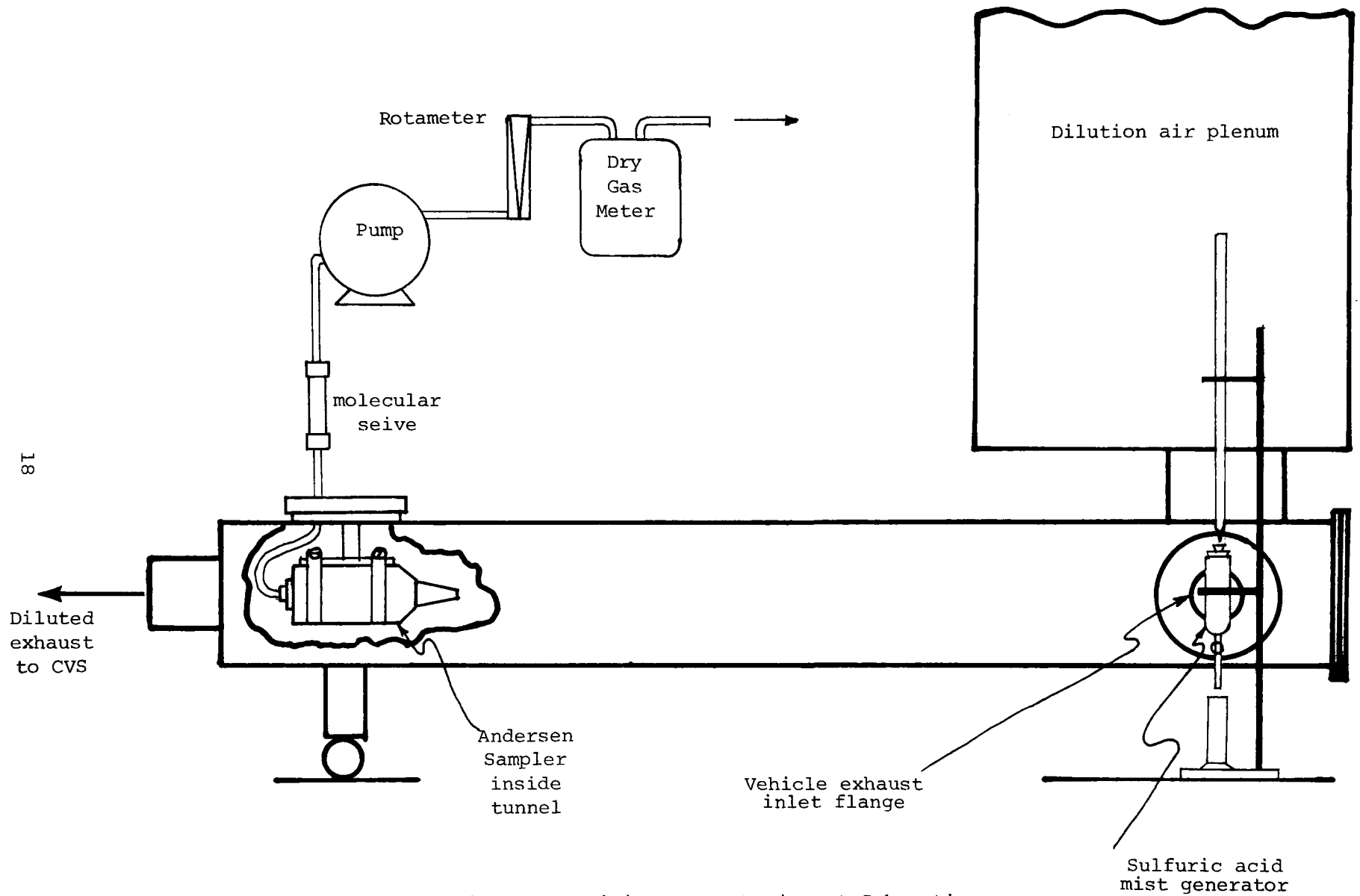


Figure 9. Sizing Tests Equipment Schematic

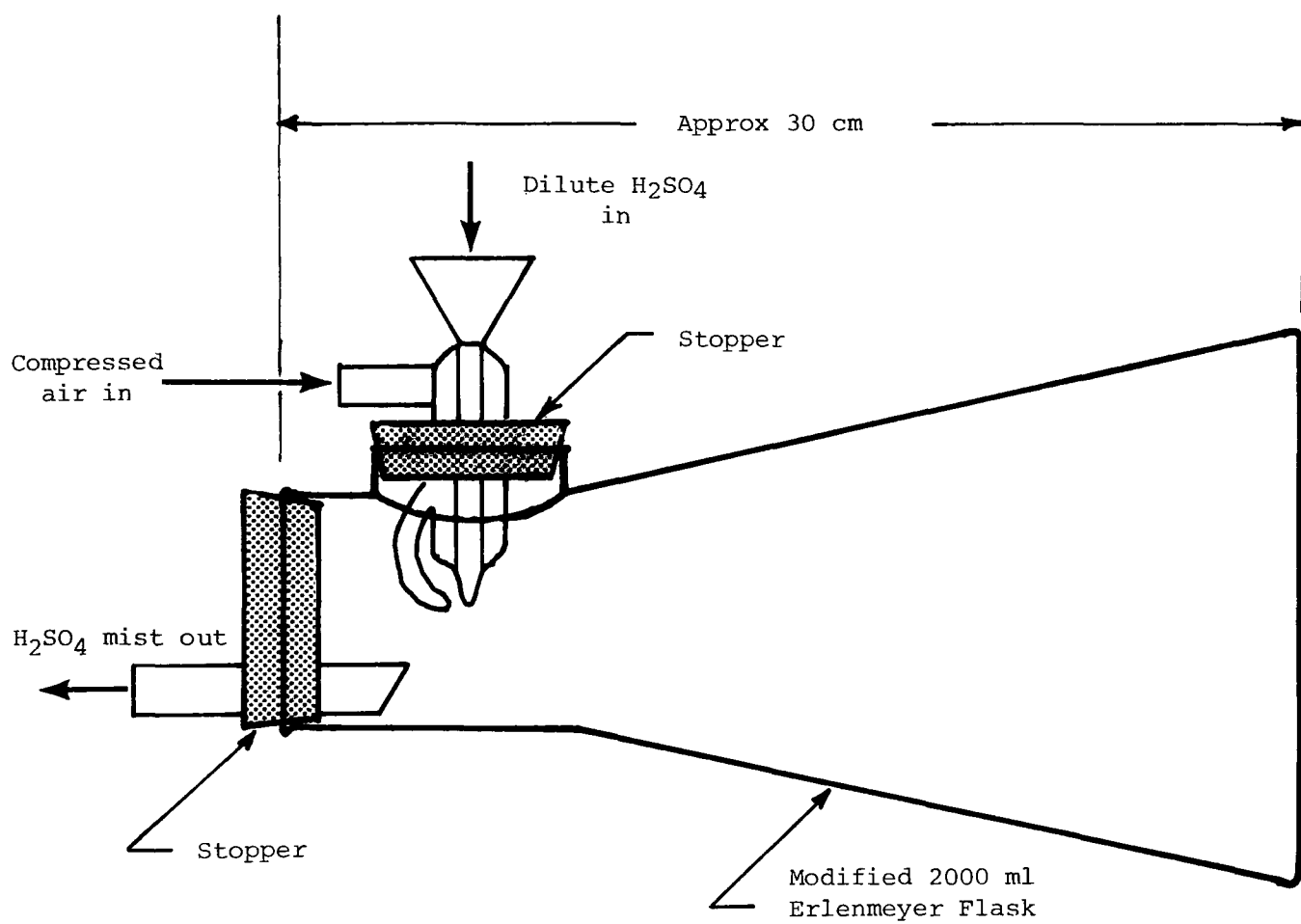


Figure 10. Modified Mist Generator

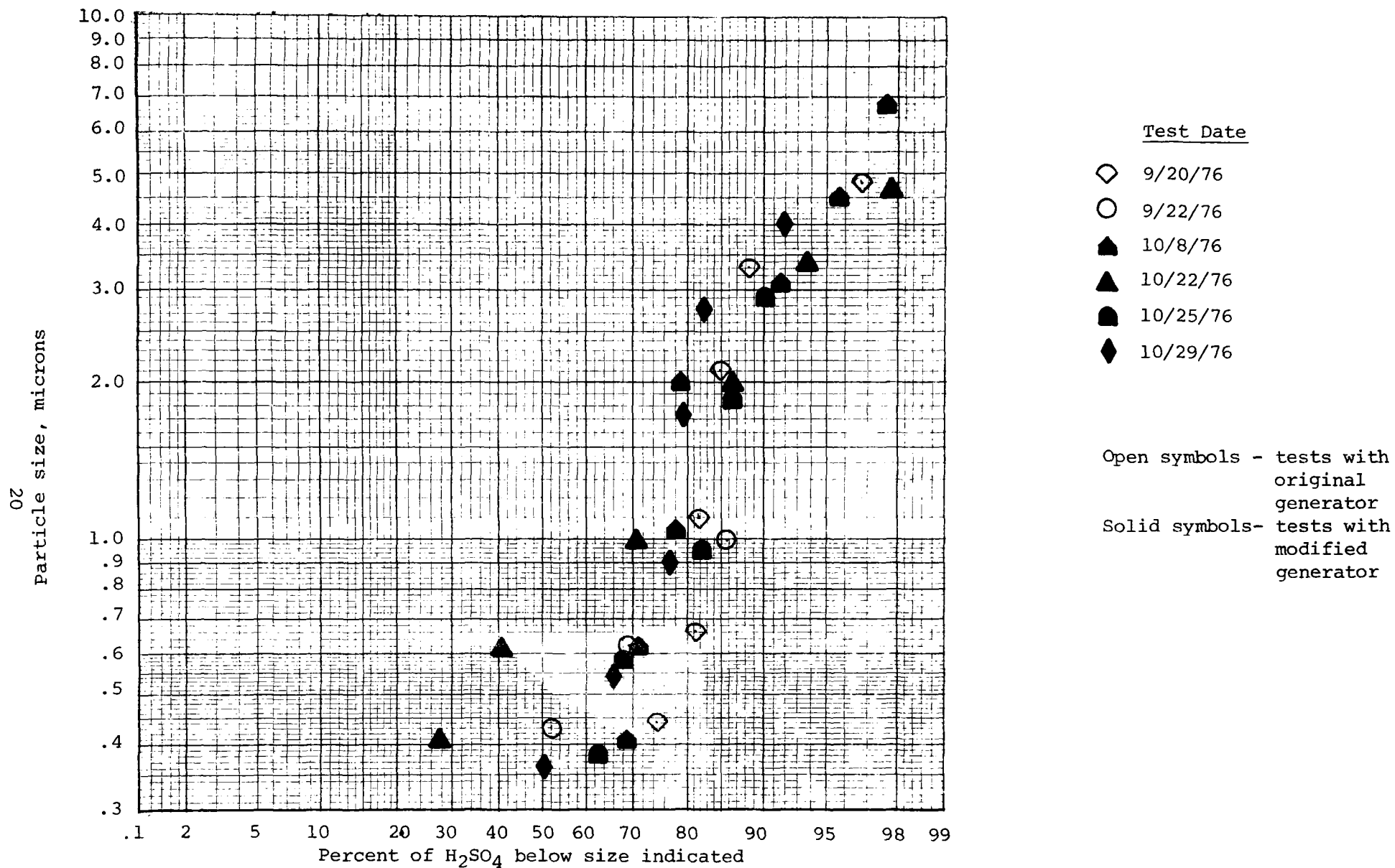


Figure 11. Results of  $H_2SO_4$  Mist Sizing Tests

than 0.36 micron. In light of this information, the Andersen Impactor has insufficient sensitivity to be used to size automotive sulfates. If it is desired to generate and measure sulfuric acid mist below 0.36 micron range, then additional development is needed on the sulfate mist generator and a different method for measuring the mist size is needed.

This test series also showed that it is extremely hard to rid the impactor plates of all residual sulfates. The time and care used to reduce the residual level of the plates for this study would be inappropriate for routine testing. For routine testing, to have sufficient sulfates deposited on the plates to make the usual residual level insignificant, either the amount of sulfuric acid put into the tunnel must be larger than can be generated by the mist generator used or the injection time must be extended to several hours.

### C. Sulfate Recovery Tests

The determination of sulfate recovery was done using the mist generator and the PDP-CVS sulfate sample probe and filter hardware. For these tests, the tunnel was heated to 53 to 57°C (128 to 135°F). The recovery tests are summarized in Table 1. The average recovery was 105 percent, with a maximum recovery of 120.8 percent and a minimum of 90.5 percent.

TABLE 1. SULFATE RECOVERY FROM MIST INJECTION TESTS

<u>Test Date</u>	<u>Average Tunnel Temp. °C</u>	<u>Test No.</u>	<u>Amount of H<sub>2</sub>SO<sub>4</sub> Injected, mg</u>	<u>Percent H<sub>2</sub>SO<sub>4</sub> Recovered on Filter</u>
10/18/76	53	1	49.0	119.0
10/18/76	54	2	71.1	94.5
10/19/76	53	1	76.0	120.8
10/26/76	57	1	64.9	101.6
10/27/76	57	1	33.1	103.6
11/3/76	56	1	60.0*	90.5
Average Recovery				105.0
Std. Dev.				12.5
Coeff. of Var.				11.9

---

\* sulfate BCA procedure used to determine amount of mist injected

The average amount of sulfate lost on the probe during the first three recovery tests was determined by washing the probe after the 10/19/76 test, (the third test), with 40 ml of 60 percent IPA and processing the solution using the BCA sulfate procedure. The amount of sulfate on the probe (in terms of µg/l of sample flow) was divided by the total sulfate (in µg/l of sample flow) from the first three tests to obtain the average percent probe loss. The probe loss for these tests averaged 3.2 percent.



Some of the spread in the recovery test data is felt to be due to the accuracy of the titration procedure for determining the amount of sulfuric acid injected into the tunnel. Due to the small amount of sulfuric acid injected into the tunnel, a difference of 0.005 in the determination of the normality of either the fresh solution injected or the remaining solution (plus washing) could make a difference on the order of 15 percentage points in recovery. The difference of 0.005 in normality translates to approximately 0.25 ml of titrant in the titration procedure. While the amount of titrant can be read to 0.1 ml, the repeatability is normally around  $\pm 0.2$  ml. To overcome this problem, a procedure was developed to use the BCA method to determine the amount of sulfuric acid injected.

The procedure is to collect the residual acid and washing, then dilute to 1000 ml total solution with distilled water. Next, a 40 ml aliquot of the solution is diluted to 100 ml with 100 percent isopropyl alcohol. This solution is then processed by the sulfate filter BCA procedure using sulfuric acid standards.

Sulfuric acid mist recovery tests using the CFV-CVS sampling system were not run, since it was felt that the pressing need was for tests with actual automotive exhaust to investigate the high total sampling system losses reported by other laboratories.<sup>(8)</sup> Thus work on the mist injection system was concluded with the results considered satisfactory for the experiments conducted. However, more development of these procedures is necessary before they could be used as a system verification test.

#### D. Tunnel Propane Traverse

A traverse of the tunnel was also performed while injecting propane into the tunnel. The results of the traverse are shown in Figure 12. There is a slight stratification from top to bottom, but it is felt that it is not large enough to affect the accuracy of the sulfate sample. The minimum and maximum concentrations were within approximately  $\pm 5$  percent of the average.

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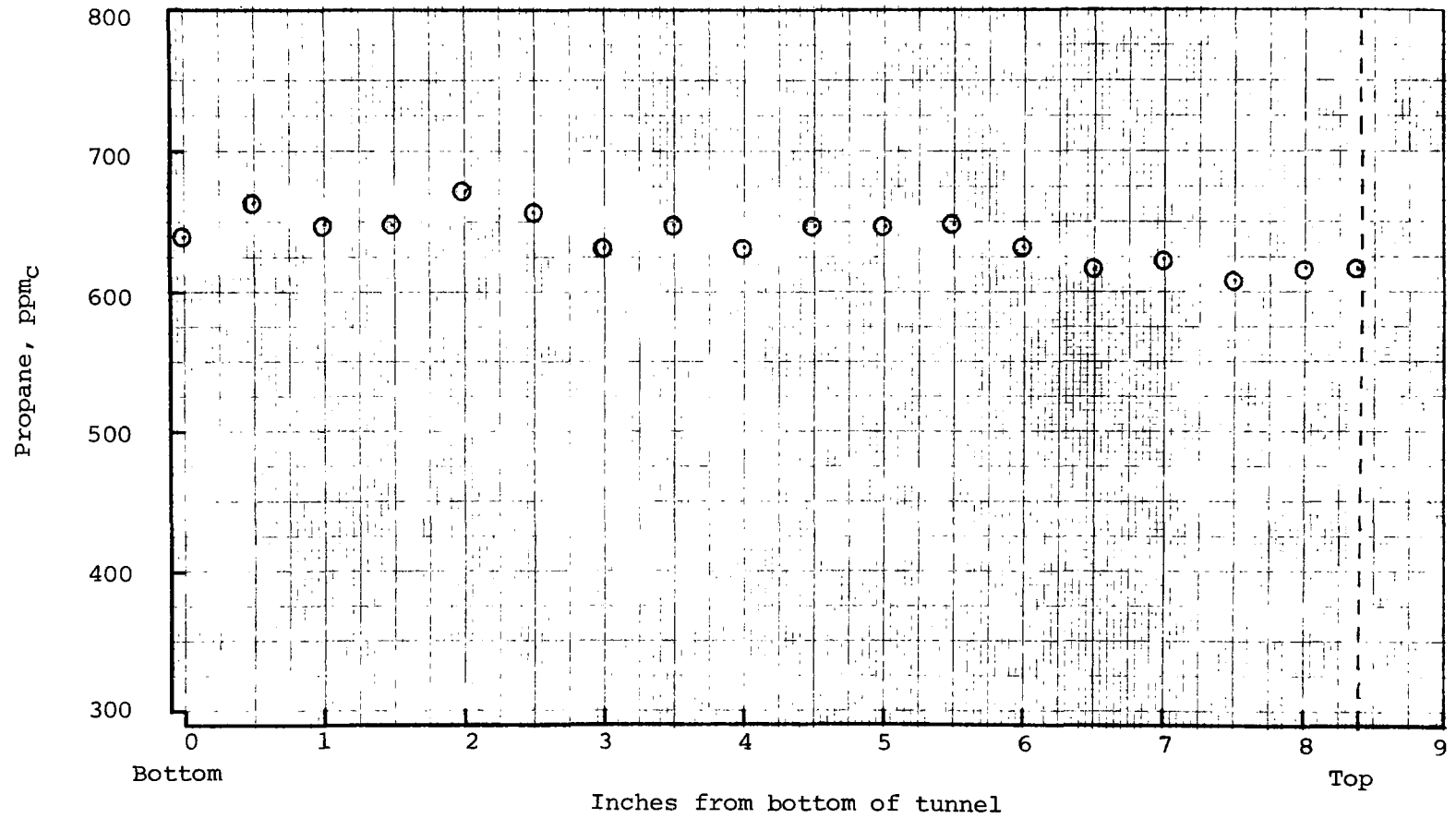


Figure 12. Tunnel Traverse During Propane Injection

#### IV. TESTS WITH AUTOMOTIVE EXHAUST

This section describes the test vehicle, procedure, and test results from sulfate tunnel tests using actual car exhaust.

##### A. Test Equipment and Procedure

The tests described in this section were performed using the 21 cm diameter tunnel described in Section II. All tests described in this section used the sample probe designed for use with PDP-CVS systems. That is, the sample probe tip did not have a choked flow section. A CVS flow rate of approximately  $9.2 \text{ m}^3/\text{min}$  (325 cfm) and a sample flow rate of  $0.023 \text{ m}^3/\text{min}$  (0.8 cfm) was used during these tests.

The test car used to generate the exhaust sulfates was a 1975 AMC Hornet Sportabout with a 4.98 litre (304 CID) V8 engine. This car was tested extensively for sulfate emissions under Contract 68-03-2118 and Task 2 of this project's Task Order Contract. The car is equipped with a pelleted catalyst and catalyst air injection system. It is known to be a higher than average sulfate producer. The fuel used was a commercially available unleaded fuel with the sulfur level adjusted to 0.04 weight percent using thiophene.

Two driving schedules were used. The first was a 64 kph (40 mph) constant speed cycle. This cycle was chosen as a condition which would produce adequate sulfates, yet would not cause undue strain on the engine, tires or dynamometer. The second cycle was the SET-7 sulfate cycle, or as it is currently called, the Congested Freeway Driving Schedule (CFDS). This cycle is a variable speed cycle with two stops and an average speed of 56.0 kph (34.8 mph). The test is 23.3 minutes long. The distance driven during the test is approximately 21.7 km (13.5 miles).

##### B. Initial Probe Loss Tests

While preparations were being made to begin comparison testing between the new tunnel and the EPA standard tunnel using actual car exhaust, the EPA Project Officer requested that some probe loss tests be run with actual car exhaust on the new tunnel. These tests were felt necessary since a General Motors study had indicated that there were considerable probe losses using a nominal 350 CFM CVS and a small (approximately 21 cm) tunnel.<sup>(8)</sup>

The tests conducted are listed in Table 2. The results of the first series of tests are shown in Table 3 under Test Series A. This test series shows that probe losses apparently can be large and seem to be at least partially dependent on the amount of sulfate drawn through the probe. The percent of total sulfate on the probe from Test Series A as a function of the amount of sulfate recovered (filter plus probe) is shown in Figure 13. For the three SET-7 tests, it appears that as the amount of sulfate put through the probe increased, the percent of sulfate on the probe decreased. Note that the tunnel temperature range for these tests was from 60°C to 100°C (140 to 212°F) with average temperature approximately 85°C (185°F).

In an effort to identify if the sulfate on the probe might be the

TABLE 2. SULFATE PROBE LOSS TESTS

<u>Date</u>	<u>SO<sub>4</sub> Probe</u>	<u>Type of Test</u>	<u>No.of Tests</u>	<u>Emissions Measured</u>
1. <u>Test Series A</u>				
12/7-8/76	1" s.s.	SET-7	10	H <sub>2</sub> SO <sub>4</sub> and gaseous (4 tests)
12/16/76	1" s.s.	SET-7	2	H <sub>2</sub> SO <sub>4</sub> and gaseous (1 test)
12/30/76	1" s.s.	SO <sub>2</sub> injection	1	H <sub>2</sub> SO <sub>4</sub> and SO <sub>2</sub>
1/7-10/77	1" s.s.	SET-7	12	H <sub>2</sub> SO <sub>4</sub> and gaseous (4 tests)
2. <u>Test Series B</u>				
1/17/77	3/8" s.s.	SET-7	3	H <sub>2</sub> SO <sub>4</sub>
1/19/77	1/4" glass	SET-7	3	H <sub>2</sub> SO <sub>4</sub>
1/19/77	1" teflon	SET-7	3	H <sub>2</sub> SO <sub>4</sub>
3. <u>Test Series C</u>				
1/26/77	1" teflon	40 mph (155°F tunnel)	1	H <sub>2</sub> SO <sub>4</sub>
1/27/77	1" teflon	40 mph (185°F tunnel)	1	H <sub>2</sub> SO <sub>4</sub>
1/28/77	1" teflon	40 mph (197°F tunnel)	1	H <sub>2</sub> SO <sub>4</sub>

Note: A 9.2 m<sup>3</sup>/min (325 cfm) PDP-CVS was used for all tests.

TABLE 3. RESULTS OF PROBE LOSS TESTS

<u>Date</u>	<u>Avg. Tunnel Temp°C</u>	<u>No. of Filters</u>	<u>Sum of SO<sub>4</sub><sup>=</sup> on Filters, µg</u>	<u>SO<sub>4</sub><sup>=</sup> on Probe, µg*</u>	<u>Total SO<sub>4</sub><sup>=</sup>, µg</u>	<u>Percent of Total SO<sub>4</sub><sup>= on Probe</sup></u>
<u>Test Series A</u>						
12/7-8/76	85	10	5178	1575	6753	23.3
12/16/76	85	2	524	577	1101	52.4
12/30/76 (SO <sub>2</sub> inj.)	50	1	>1	23	----	----
1/7-10/77	85	12	4257	1226	5483	22.3
<u>Test Series B</u>						
1/17/77 (3/8" s.s.)	85	3	700	426	1125	37.8
1/19/77 (1/4" glass)	85	3	813	341	1154	29.6
1/19/77 (1" Teflon)	85	3	1521	542	2063	26.3
<u>Test Series C</u>						
1/26/77	68	1	1963	69	2032	3.4
1/27/77	85	1	2214	221	2435	9.1
1/28/77	92	1	1595	679	2274	29.9

Note that the results for each line represent the total aggregate sulfates from a number of repetitive tests.

\*From single analysis at end of each set of repetitive tests.

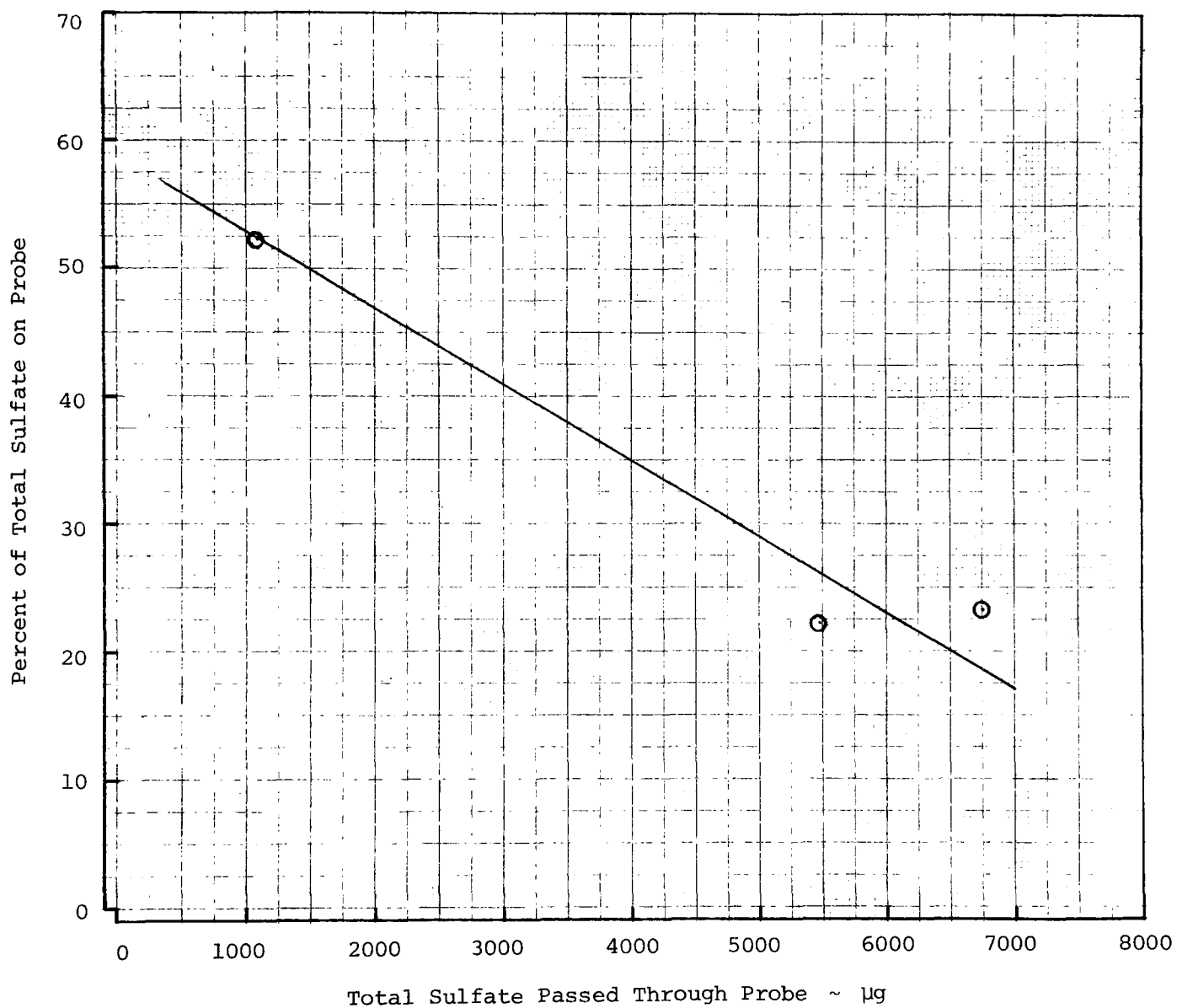


Figure 13. Percent Probe Loss Versus Sulfate Through Probe

result of a reaction of the probe wall with sulfur dioxide, (SO<sub>2</sub>), a test was run injecting pure SO<sub>2</sub> into the tunnel. The SO<sub>2</sub> was injected at a rate approximately equal to exhaust SO<sub>2</sub> during SET-7 test. This test was run at a tunnel temperature of 50°C (122°F) which is close to the maximum that can be obtained with the tunnel heaters without automotive exhaust being put into the tunnel. The sulfate found on the filter was equivalent to the usual background level. Sulfate above residual levels was found on the probe. However, the sulfate level was less than a tenth of the sulfate levels seen from the two previous probe loss tests run with car exhaust.

At the time the test was run, the results of the SO<sub>2</sub> injection test were interpreted to mean that the probe sulfate did not come from a reaction of the probe walls with SO<sub>2</sub>. Later tests with car exhaust showed the probe losses to be a function of temperature as will be explained below. While no tests with car exhaust were run at tunnel temperatures as low as 50°C, the trend from Figure 15 indicates that probe losses with actual car exhaust could be the same at 50°C as that seen from the SO<sub>2</sub> injection test. Thus it is not certain how the results of the SO<sub>2</sub> injection test should be interpreted. Therefore, SO<sub>2</sub> injection tests over a range of tunnel temperatures are recommended as a possibility for future investigations. One way to do this is to use sulfur sterile fuel during tests with an actual car and inject SO<sub>2</sub> into the exhaust at the tunnel entrance.

A second series of tests was run to determine if probe material affected the probe losses. This series of tests is listed in Tables 2 and 3 as Test Series B. In this series, three consecutive SET-7 tests using the 1975 Hornet were run with each of three types of probes. These tests indicate that while probe material did have an effect on probe losses, substantial losses can occur using stainless steel, glass and Teflon probes at tunnel temperatures of 85°C (185°F). The 9.5 mm (3/8 inch) stainless steel probe had the highest losses, 37.8 percent, and the 2.54 cm (1 inch) Teflon lined probe the smallest losses, 26.3 percent.

Three tests were run at different tunnel temperatures to investigate the effect of temperature on probe losses. The test series is listed in Tables 2 and 3 as Test Series C. Each test consisted of a 64 kph steady state run for one-half hour using the 1975 Hornet. These tests show that tunnel temperature does affect probe losses, with the probe losses increasing from 3.4 percent at 68°C (155°F) to 29.9 percent at 92°C (197°F).

#### C. System Sulfate Losses as a Function of Temperature

The initial series of tests showed that substantial probe losses can occur with stainless steel, glass and Teflon probes and that the probe losses were a function of the amount of sulfate passed through the probe and the tunnel temperature. Since there was such a large difference in the probe loss between 85°C (185°F) and 92°C (197°F), it was felt by both EPA and SwRI that further testing was necessary to verify the shape of the probe loss curve with temperature. This test series is listed in Table 4.

A series of six tests were run using the 1975 Hornet as a source of exhaust

TABLE 4. TUNNEL TEMPERATURE EFFECTS TEST SCHEDULE

<u>Test</u>	<u>Tunnel Temp., °C</u>	<u>Test Condition</u>	<u>Probe</u>	<u>Emissions Measured</u>
1	68	1/2 hr at 40 mph	1" s.s.	SO <sub>4</sub> , SO <sub>2</sub> , gaseous
2	77	1/2 hr at 40 mph	1" s.s.	SO <sub>4</sub> , SO <sub>2</sub> , gaseous
3	85	1/2 hr at 40 mph	1" s.s.	SO <sub>4</sub> , SO <sub>2</sub> , gaseous
4	88	1/2 hr at 40 mph	1" s.s.	SO <sub>4</sub> , SO <sub>2</sub> , gaseous
5	91	1/2 hr at 40 mph	1" s.s.	SO <sub>4</sub> , SO <sub>2</sub> , gaseous
6	96	1/2 hr at 40 mph	1" s.s.	SO <sub>4</sub> , SO <sub>2</sub> , gaseous

Note: Both the probe and the tunnel were washed with 60 percent IPA following each test.

TABLE 5. SUMMARY OF SYSTEM SULFATE LOSSES

<u>Test</u>	<u>Filter SO<sub>4</sub><sup>=</sup> µg/m<sup>3</sup></u>	<u>Sulfate Losses, µg/m<sup>3</sup> @ 9.20 m/min. CVS Flow Rate</u>						<u>Total Sulfates µg/m<sup>3</sup></u>	<u>Tunnel Temp °C</u>
		<u>Probe</u>	<u>Tunnel</u>	<u>Elbow</u>	<u>0.46 m. Flex pipe</u>	<u>Tunnel + elbow + flex</u>	<u>Total losses</u>		
1	3052.61	59.32	52.36*	12.08	28.60	92.94	152.26	3204.87	68
2	3002.47	72.03	57.66	9.04	29.59	96.29	168.32	3170.79	77
3	2496.82	175.85	67.97	5.12	39.09	112.18	288.03	2784.96	85
4	2673.02	264.12	101.69	5.76	29.03	136.48	400.60	3073.80	88
5	2843.57	271.89	116.14	8.16	31.78	156.08	427.97	3271.54	91
6	2457.98	548.38	129.84	6.71	30.90	167.45	715.83	3173.73	96

Percent of Total Sulfate

1	95.39	1.85	1.63	0.38	0.79	2.80	4.65		68
2	94.67	2.27	1.82	0.29	0.93	3.04	5.31		77
3	89.65	6.31	2.44	0.18	1.40	4.02	10.33		85
4	86.97	8.59	3.31	0.19	0.94	4.44	13.03		88
5	86.92	8.31	3.55	0.25	0.97	4.77	13.08		91
6	77.45	17.28	4.09	0.21	0.97	5.27	22.55		96

\*estimate from curve of tunnel temperature vs sulfate loss



sulfate to determine the sulfate losses in the sampling system with changes in tunnel temperature. A Teflon lined probe was used during these tests. Each test consisted of a 25 minute run at 40 mph. An initial acceleration from idle to 40 mph was part of each test. At the conclusion of the test, the sulfate level in the sample probe and in all system components upstream of the probe was determined.

Table 5 lists the sulfate on the filters together with the sulfate found on each of the elements of the sample system. In the top half of the table, the sulfate levels are expressed in micrograms per actual cubic meter of dilute exhaust flow. The bottom part of the table shows the sulfate levels as a percent of the total sulfate. As can be seen from the table, the probe and tunnel losses increase as the tunnel temperature increases. The flexible tubing connector losses measured for each test are constant, as would be expected, at about 1 percent of the total sulfate. Figures 14 and 15 show the percent probe and tunnel losses as a function of tunnel temperature. Figure 16 shows the total sulfate losses (probe, tunnel, elbow, and flex) as a function of tunnel temperature.

It should be noted that these results are from one set of tests on a particular car. It may be that the sampling system losses are also a function of the sulfate concentration. Since the car used on this project has much higher than average sulfate emissions, the sulfate losses shown here may not be typical.

Remember also that the probe loss tests covered in the previous section seem to indicate that a stainless steel probe would give somewhat higher losses than a Teflon probe. Those tests also indicated that there is a "seasoning" effect on the sampling system and that the losses could go down considerably after a certain number of tests if the system was not washed after every test. Thus while a considerable amount has been learned about the sulfate sampling system losses, there are still many areas where more information is needed.

#### D. Effect of Connecting Tubing Length

The various laboratories engaged in automotive sulfate sampling use different lengths of flexible exhaust tubing to connect the car exhaust system to the sulfate tunnel. One of the items of investigation in this study was to evaluate the effects of this connecting tubing length on sulfate losses. Two different lengths of 10.2 cm (4 inch) diameter tubing were used, a 0.61 m (2 foot) length and a 4.6 m (15 foot) length.

Several tests, each 25 minutes in length, at 64 kph (40 mph) steady state were run with each length of connecting tubing. The test results are summarized in Table 6. For these tests, probe and tunnel losses taken from Figures 14 and 15 were used together with the sample filter sulfate value from each test to obtain the total sulfate level.

As can be seen from the table, the sulfate losses in the connecting tubing increased considerably as the tubing length increased. For the 0.61 m (2 foot) length, the loss was 0.6 percent and for the 4.6 m (15 foot) length, the loss was 20.4 percent.

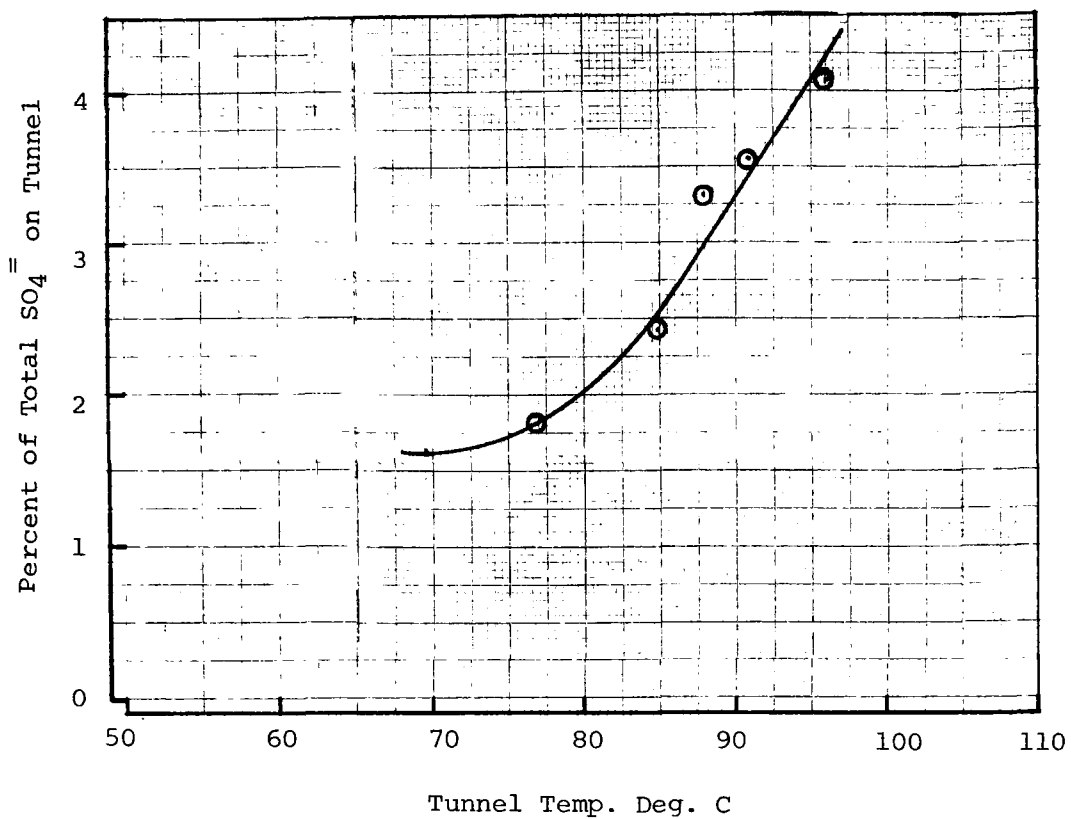


Figure 14. Percent  $\text{SO}_4$  on Tunnel Versus Tunnel Temperature

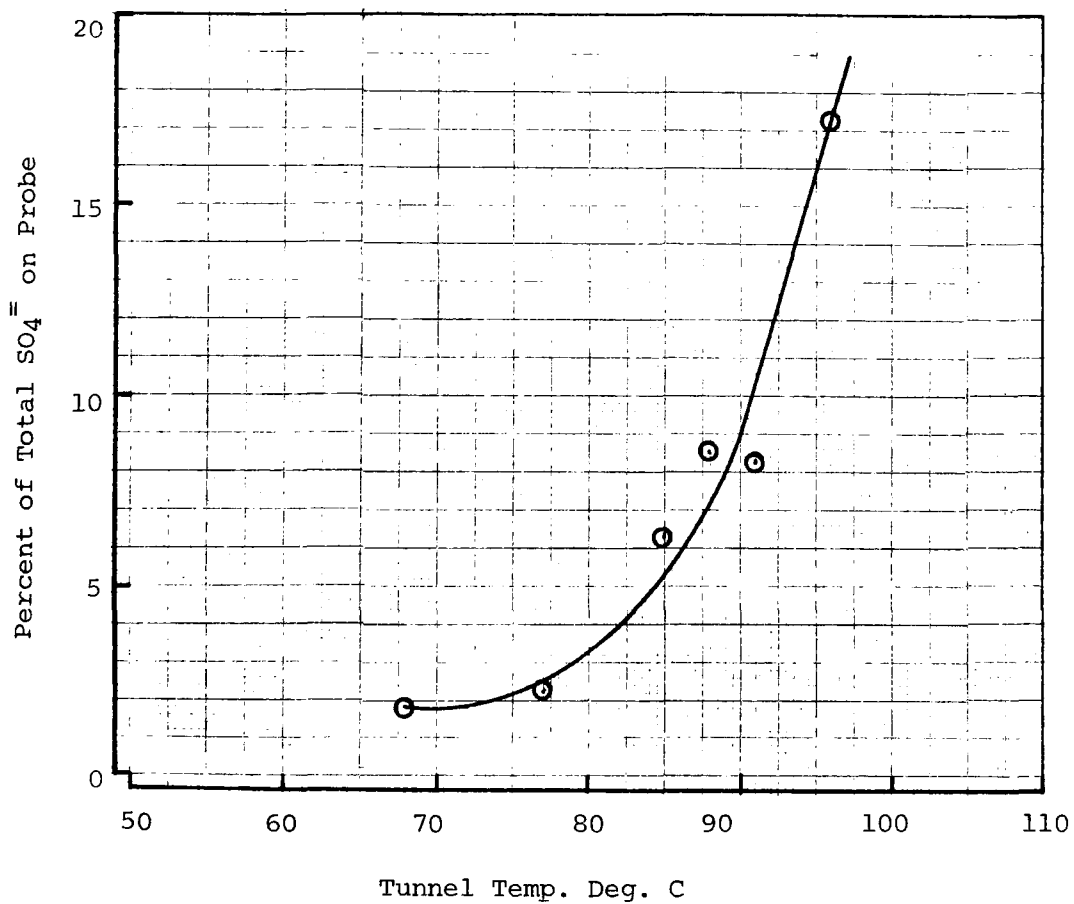


Figure 15. Percent  $\text{SO}_4$  on Probe Versus Tunnel Temperature

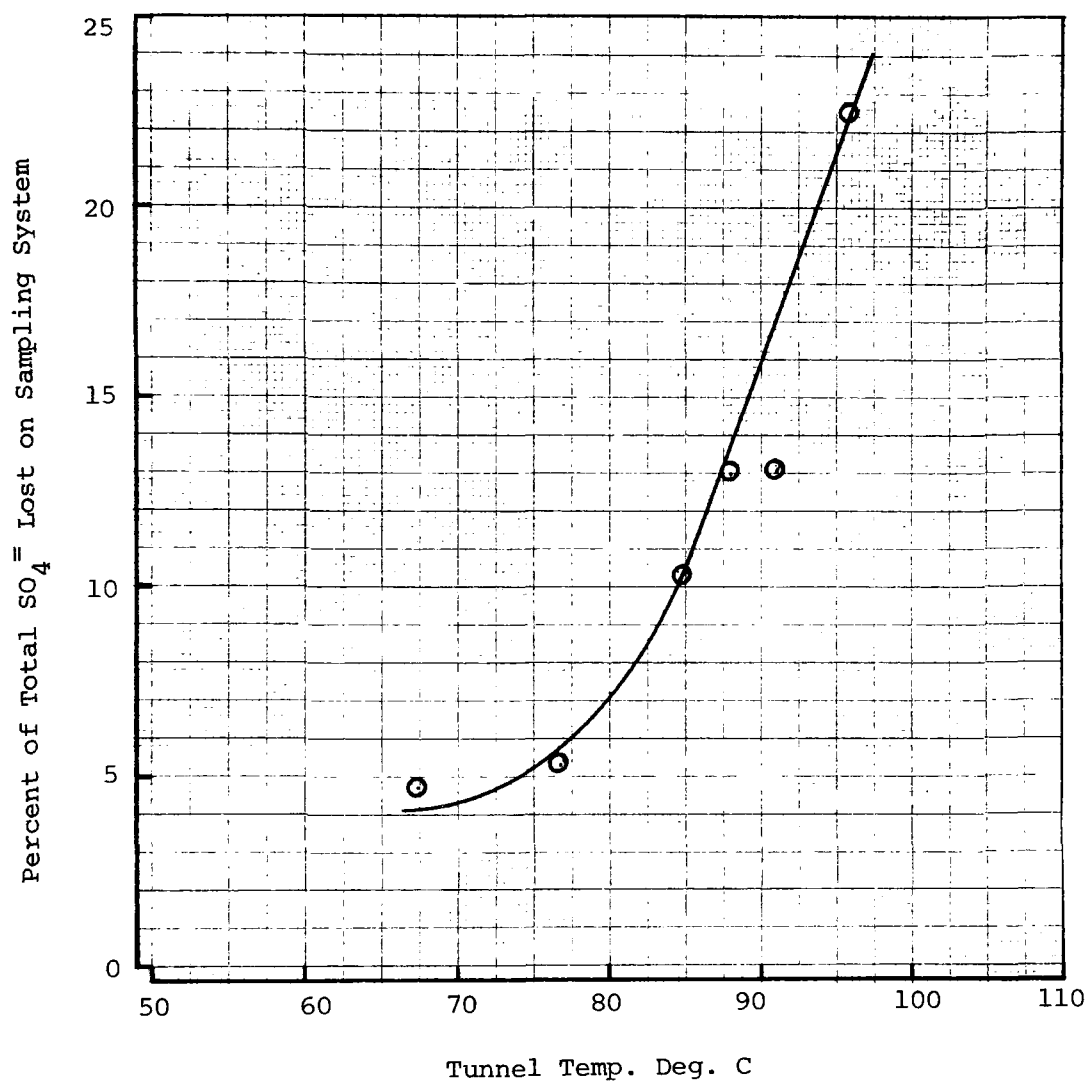


Figure 16. Percent  $\text{SO}_4^{=}$  Lost on Sulfate Sampling System  
Versus Tunnel Temperature

TABLE 6. EFFECTS OF CONNECTING TUBING LENGTH

<u>Test Date</u>	<u>Flexible Pipe Length</u>	<u>Vehicle Exhaust Temp °C</u>	<u>Exhaust temp into Tunnel °C</u>	<u>Tunnel Temp °C</u>	<u>Percent of total SO<sub>4</sub> on flex pipe</u>	<u>Total SO<sub>4</sub> mg/km</u>
3/31/77	0.61 m (2 ft)	263	224	66.7	0.63	37.59
4/13/77	0.61 m (2 ft)			66.7	<u>0.51</u>	40.15
					Avg 0.57	
3/31/77	4.6 m (15 ft)	254	163	49.4	20.0	42.54
4/13/77	4.6 m (15 ft)			53.9	23.4	38.20
4/15/77	4.6 m (15 ft)			66.7*	<u>17.8</u>	41.87
					Avg 20.4	

\*Tunnel dilution air preheated to maintain tunnel temperature

## V. SYSTEM MODIFICATIONS

As a result of the testing explained in the previous sections of this report as well as some additional tests reported in this section, some system modifications were required. This section explains the rationale for those changes.

### A. Critical Flow Sample Rate Evaluation

A series of tests were conducted to check out the components of the sulfate sampling system designed for use with a CFV-CVS. Recall that a critical flow section in the sampling system is required for the CFV-CVS and that the probe entrance nozzle was chosen as the location for this critical flow (choked) section. For choked flow the static pressure at the nozzle throat must be 0.528 of the nozzle inlet static pressure.<sup>(5)</sup> Although some pressure is recovered in the diverging section of the nozzle, to insure choked flow it was planned to keep the pressure downstream of the nozzle at approximately 0.5 of the nozzle inlet pressure. Since the pressure drop across the filter is large, it was recognized from the start that a sample pump with high vacuum capability would be needed.

Prior to obtaining a pump, the nozzle was calibrated using ambient air. The calibration curve is shown in Figure 17. The pressure loss curve for a clean 0.5 micron Fluoropore filter was also determined and is presented in Figure 18. If a tunnel pressure of approximately 735 mm of mercury is assumed, then from Figure 17 and 18 it can be determined that a sample pump capable of 0.028 standard m<sup>3</sup>/min (1 scfm) flow at 636 mm (25.0 inches) of mercury vacuum is required. After a considerable search, a pump was located with this capability. The pump is a Gask Model 2565. The manufacturers published flow ratings are shown in curve form in Figure 19.

A SET-7 test was run with the CFV-CFS sample probe using a car known to be a high sulfate producer (on the order of 50 mg/km). As the filter loaded during the test, its pressure losses increased. At approximately 1100 seconds into the test, the pressure loss became sufficient that the nozzle unchoked. The flow continued to drop for the remainder of the test. From this test it was concluded that 0.028 m<sup>3</sup>/min (1.0 cfm) sample rate was too large to maintain choked flow with a 47 mm, 0.5 μm Fluoropore filter. After discussing the problem with the Project Officer, it was decided to set the sample flow at 0.014 m<sup>3</sup>/min (0.5 cfm).

### B. Methods to Reduce Tunnel Temperature

The tests discussed in the previous section demonstrated that sulfate losses on the probe and tunnel increase as the tunnel temperature increased. This section briefly examines two methods of lowering the sulfate tunnel temperature and presents the modification made to arrive at the final tunnel configuration. However, since the elevated tunnel temperature is caused by the hot vehicle exhaust gas, it was first necessary to determine what temperature should be used as exhaust gas temperature in a system design.

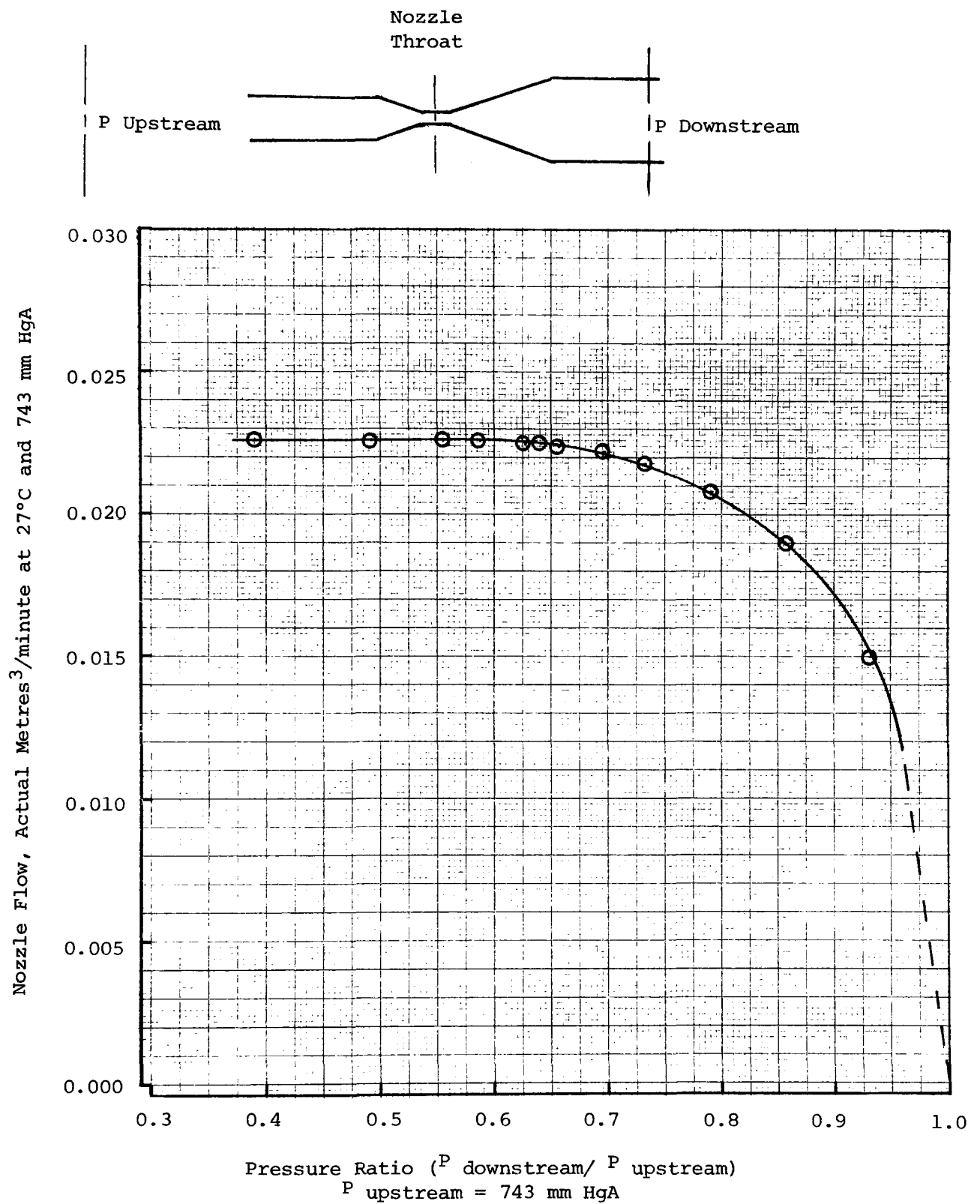


Figure 17. Calibration of SwRI Built Critical Flow Nozzle

— least squares straight line  
 $Y = 0.00110 + (7.8307 \times 10^{-5})(X)$   
 $r^2 = 0.997$

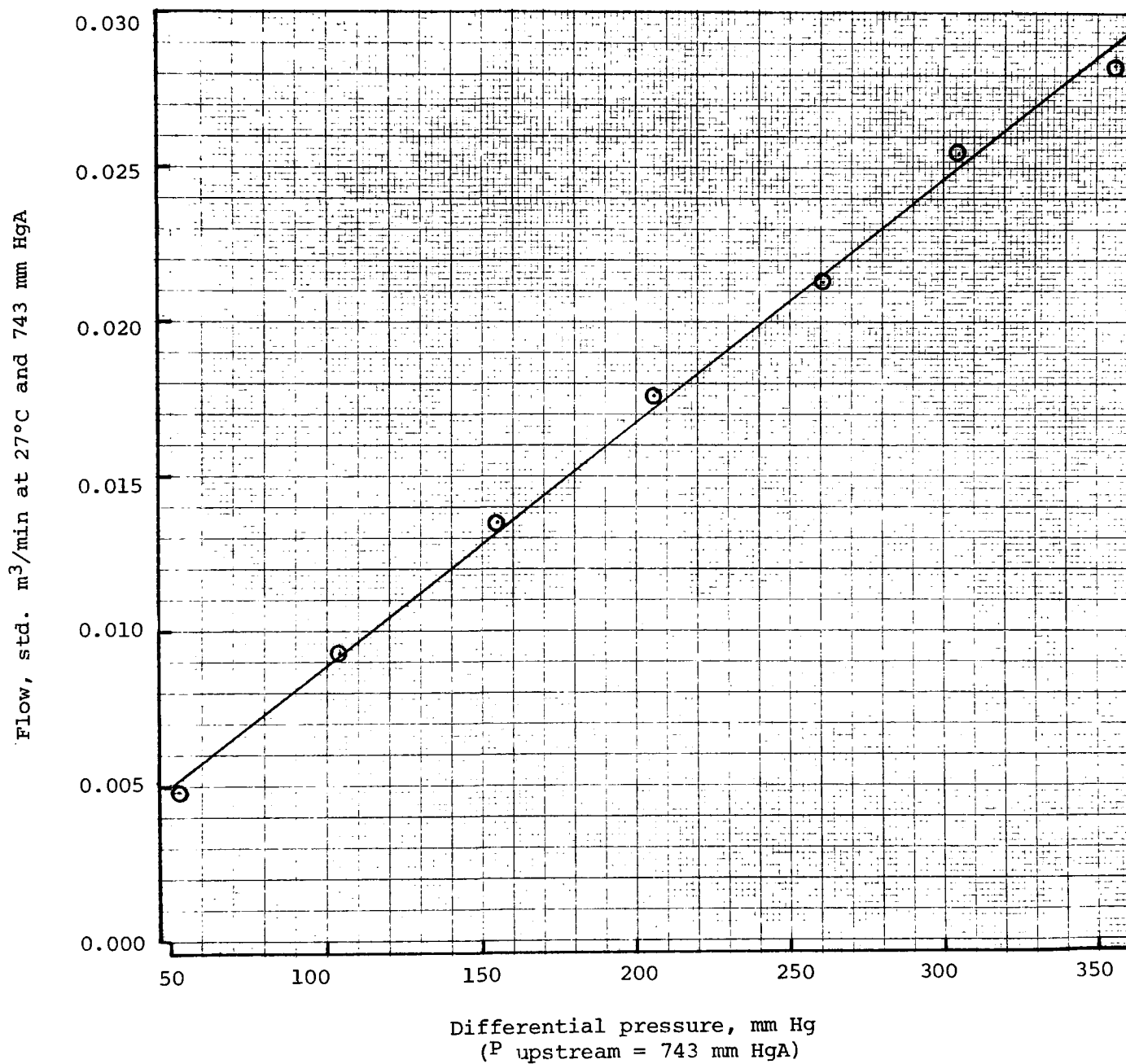


Figure 18. Pressure Drop Across Clean 47 mm Fluoropore Filter

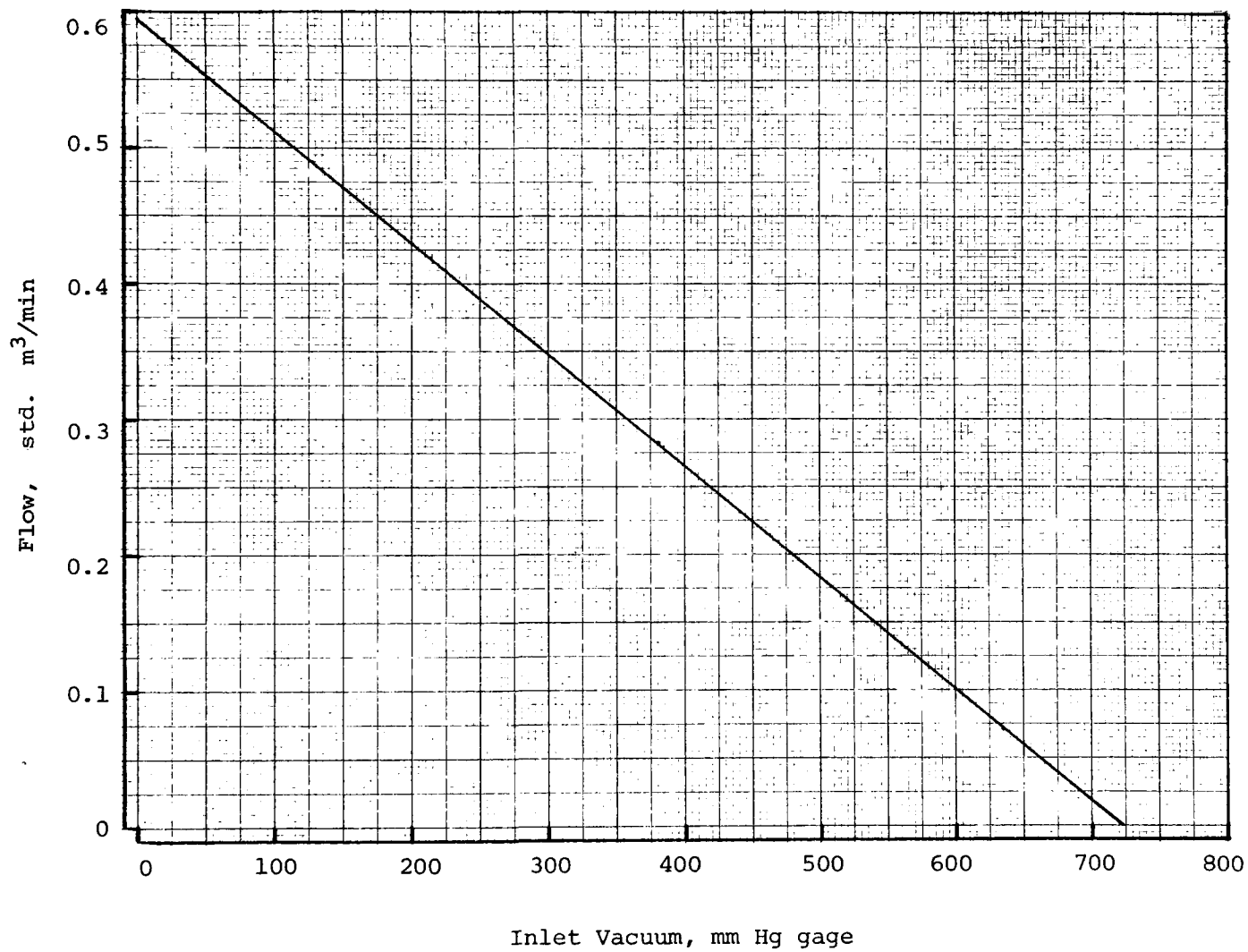


Figure 19. Capacity Curve for Gast 2565 Pump



The exhaust gas temperature of three different catalyst cars were measured during dynamometer operation. Table 7 shows the maximum exhaust temperature obtained on these cars.

While this is certainly not a large sample, it does show that exhaust temperatures on the order of 380°C (716°F) are possible during the SET-7. It is probable that other vehicles could have higher exhaust temperatures. Nevertheless, for this analysis, 380°C was used for a design exhaust gas temperature.

TABLE 7. MAXIMUM EXHAUST TEMPERATURE DURING  
DYNAMOMETER OPERATION FOR THREE CARS

<u>Car</u>	<u>Mfgr.</u>	<u>Engine Size, liters</u>	<u>Inertia, kg</u>	<u>Cycle Type</u>	<u>Max. Exhaust Temp. °C</u>
1	Ford, LTD	5.75	2268	FTP	285
1	Ford, LTD	5.75	2268	FET	350
2	Chevrolet	5.74	2041	SET-7	378
3	Hornet	4.98	1587	SET-7	356

#### 1. Cooling Dilution Air

One method for lowering the tunnel temperature is to cool the dilution air. For a simple air conditioning system, the cooling limit for the dilution air should be about 4°C (39°F) to prevent ice formation on the coils. Assuming 24°C (75°F) room air, this is about a 20°C (36°F) drop in dilution air temperature. Cooling the dilution air to 4°C results in a tunnel temperature drop of about 14°C (26°F) using a nominal 9.20 m<sup>3</sup>/min (325 CFM) CVS with a blower inlet cooled to 43°C (110°F) and a 5.74 liter (350 CID) engine with exhaust temperature of 380°C (716°F). If it is desired to keep the tunnel below 77°C (170°F), this amount of cooling would not be sufficient for a 5.74 liter engine in a 2041 kg car.

With a more complex cooling system, the air could be cooled to lower temperatures. Using the example 5.74 liter car from above, a dilution air temperature of about -35°C (-32°F) is required to lower the peak tunnel temperature to 77°C (170°F). This low temperature could also cause problems with condensation.

The above examples assume a CVS with a heat exchanger to maintain the blower inlet temperature at about 43°C (110°F). If a CFV-CVS without a heat exchanger is used, more cooling would be required since as the tunnel temperature increases, mass flow through the CFV-CVS decreases, causing the tunnel temperature to rise. Also, sophisticated temperature control is required due to the rapidly changing exhaust temperature and flow rate.

## 2. Increase Amount of Dilution Air

Another method for lowering the tunnel temperature is to increase the CVS capacity, thus increasing the dilution air flow. Using the 5.74 liter car from the example above, a CVS with approximately 17.0 m<sup>3</sup>/min (600 CFM) capacity would be required to lower the peak tunnel temperature to 77°C (170°F). A CFV-CVS without a heat exchanger would have to be somewhat larger, on the order of 18.5 m<sup>3</sup>/min (650 CFM) for reasons explained above.

## C. Final System Modification Chosen

After considering all the factors involved, it was decided by the EPA Task Officer that the small (21 cm) tunnel should be used together with a larger CVS. It was requested that the sampling system nozzle be sized for 525 cfm CVS flow and 0.5 cfm sample flow. The new nozzles were made and a series of three tests run on the completed sulfate sampling system with the critical flow nozzle and a 520 CFM CVS. The tests run are described in Table 8. The results are shown in Table 9.

From the Table 9, it is evident that the increased flow rate reduced the sulfate losses on the tunnel walls. The sulfate losses in the flexible connecting tubing were slightly higher than seen in previous tests with lower CVS flow, while the losses in the exhaust entrance elbow were about the same. The sulfate losses in critical flow sample probe were considerably higher than seen in tests with the noncritical flow probe and lower CVS flows.

The probe losses of 15 and 22 percent from the tests 1 and 2, with average temperatures of 69°C (156°F) and 74°C (165°F) respectively, are higher than desired for routine sulfate testing. For the noncritical flow probe at temperatures below 77°C, the probe losses were less than 3 percent. While time did not permit further investigation of the sulfate losses with the critical flow probe, it is recommended that a thorough investigation of the losses with this probe be conducted prior to using this system for routine sulfate testing.

TABLE 8. TEST SCHEDULE WITH LARGE CVS

<u>Test No.</u>	<u>Test Type</u>	<u>Duration (min)</u>	<u>heater condition</u>
1	64 kph steady	30	Max heat
2	SET-7	23.3	Max heat
3	SET-7	23.3	Min. temp controlled to 43°C (110°F)
<u>For All Tests</u>			

Notes: 1. CVS flow 14.7 m<sup>3</sup>/min (520 cfm)  
 2. critical flow nozzle used on probe  
 3. sample flow = 0.014 m<sup>3</sup>/min (0.5 cfm)

TABLE 9. RESULTS FROM TESTS WITH LARGE CVS

<u>Test No.</u>	<u>Test Type</u>	<u>Average Tunnel Temp. °C</u>	<u>Filter Sulfate <math>\mu\text{g}/\text{m}^3</math></u>	<u>Sulfate Losses <math>\mu\text{g}/\text{m}^3</math></u>					<u>Total Sulfates <math>\mu\text{g}/\text{m}^3</math></u>
				<u>CFV Probe</u>	<u>Tunnel</u>	<u>Elbow</u>	<u>Flex Pipe</u>	<u>Total Losses</u>	
1	64 kph	69	1624.82	307.80	10.76	7.20	51.23	376.99	2001.81
2	SET-7	74	2077.29	615.45	9.60	12.86	60.13	698.04	2775.33
3	SET-7	59	1902.15	125.67	3.64	12.43	43.15	184.89	2087.04

<u>Test No.</u>	<u>Test Type</u>	<u>Tunnel Temp. °C</u>	<u>Percent of Total Sulfate</u>					
			<u>Filter</u>	<u>CFV Probe</u>	<u>Tunnel</u>	<u>Elbow</u>	<u>Flex Pipe</u>	<u>Total Losses</u>
1	64 kph	69	81.16	15.38	0.54	0.36	2.56	18.84
2	SET-7	74	74.85	22.18	0.35	0.46	2.17	25.16
3	SET-7	59	91.14	6.02	0.17	0.60	2.07	8.86

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7. Wilson, W. E., et al, "General Motors Sulfate Dispersion Experiment: Summary of EPA Measurements." Journal of the Air Pollution Control Association, Vol. 27, No. 1, January, 1977.
8. Letter from C.J. Elder, Environmental Activities Staff, General Motors Corporation, to Richard Lawrence, Emission Control Technology Division, EPA, Ann Arbor, Michigan, dated October 26, 1976.

APPENDIX A  
STATEMENT OF WORK

STATEMENT OF WORK

The purpose of this task is to provide EPA with a dilution tunnel and sampling system compatible with the critical flow venturi CVS units used in certification testing.

1. Design and build a dilution tunnel and sampling system which will meet the following requirements:

- (a) Provide mixing of the exhaust and dilution air.
- (b) Be compatible with a CVS flow rate of 325 SCFM.  
A typical dilution tunnel operating conditions will be 735 mm Hg. A and 150F.
- (c) Be compatible with EPA certification test cells, as determined by discussions with project officer or other persons designated by project officer.

2. Design parameters such as dilution tunnel length, diameter, material and configuration; mixing orifice diameter; probe location, material and size; and sampling train components and configuration will be determined by discussion with the task officer and others, as appropriate. These parameters should be chosen within the first 10 days of the task period.

3. Two planning meetings have been held in Ann Arbor and the following parameters have been tentatively agreed upon:

- (a) The dilution tunnel diameter will be approximately 8 inches.
- (b) The distance between the exhaust inlet and the probe tip will be 8 pipe diameters.
- (c) The distance between the probe tip and the downstream end of the tunnel will be two pipe diameters.
- (d) The tunnel material will be stainless steel.
- (e) A mixing orifice will be located at the plane of the exhaust inlet.
- (f) The probe will be made of stainless steel tubing.
- (g) The filter holder will be outside the tunnel

4. Design and build a sampling system compatible with critical flow venturi CVS units. Flow velocity into the probe shall be within  $\pm 10\%$  of the velocity of the dilute exhaust mixture adjacent to the probe at all times. Sampling through a critical flow venturi or other means such as electronic control can be used to accomplish the desired flow control.

5. Design and build an apparatus which will inject a known amount of sulfuric acid aerosol into the dilution tunnel. Dilution tunnel and analytical system verification can be performed using this apparatus in much the same way a propane injection is currently used for CVS

ification. The size of the aerosol should be measured and 98% <sup>of the aerosol mass</sup> should be less than one micron aerodynamic diameter. It is recognized that the development work specified in this item will not necessarily result in a good sulfuric acid mass injection system. A "best effort" is expected. Problem areas should be identified so that subsequent investigations can address them.

6. Validate the tunnel and sampling system by running the following tests:

- (a) Sulfuric acid injection using the apparatus developed in 5, above, if possible.
- (b) Continuously inject propane while performing radial traverses with a probe in the vicinity of the sulfate probe location. Record HC concentration continuously. Traverses in 2 radial directions should be performed. Plot HC concentration versus radial distance. Determine the difference between the average observed HC concentration and the expected value.

7.\* Compare the new tunnel at approximately 300 SCFM with the 18" tunnel (on hand at SwRI) at 475 to 600 SCFM by:

- (a) Alternating vehicle exhaust from a vehicle at steady cruise conditions between the two systems. Approximately 20 samples ( $H_2SO_4 + SO_2 +$  gaseous emissions) will be taken in each configuration. Since a CFV-CVS is not available at SwRI, a standard PDP-CVS can be used with the new tunnel, however the new CFV compatible sample system should be used for these steady state tests. During the 300 CFM tests both the CFV sampling system and a standard probe should be operated in parallel. Sulfuric acid level and probe losses determined with each system can then be compared.
- (b) Running consecutive sulfate cycles, alternating the vehicle exhaust from one system to the other. Approximately six cycles with each tunnel will be required. A standard probe and sampling system may be required if a PDP-CVS is used instead of a CFVCVS.

Determine probe and tunnel losses with each system. A single determination for all tests run in each tunnel in each of parts (a) and (b) above is sufficient. Continuously record the temperature of the sample entering the filter holder. Note that the CFV sampler is designed for 325 SCFM but that operation at the low end of the normal CVS range, i.e. 300 SCFM, is requested here. This should not cause a problem since these two flows are within 10% of each other, which is the range allowed in item 4, above.

8. Determine the effect of the length of connecting tubing from vehicle to tunnel (tubing specifications to be supplied by EPA). Record temperature of exhaust gas at vehicle tailpipe and dilution tunnel inlet using both short (2') and long (15') lengths of tubing. Measure sulfuric acid trapped in the connecting tubing. The following method, or equivalent, should be used.

- (a) Rinse the tubing with a 60/40 IPA solution until an analysis of the IPA shows little or no sulfate response on the barium chloranilate (BACL) analytical system.
- (b) Run one or more emission tests (S7).
- (c) Rinse the tubing with IPA and analyze the rinse solution for sulfate.

9. \*Determine the effect of dilution air humidity by running S-7 tests at three dilution air humidity levels of 100 to 110, 70 to 80, and 20 to 40 grains/pound. Measure probe losses and size distribution of the collected particulate.

10. Deliver the dilution tunnel and sampling system to EPA in Ann Arbor, Michigan. Provide technical assistance in setting up and checking out the complete system with a CFV-CVS. For planning purposes an estimate of 1 man for 3 days at the Ann Arbor facility is reasonable.

11. Provide 3 complete sets of shop drawings and specifications, including one reproducible set.

12. Other.

- (a) The sulfate analytical system should be dedicated to this test work so that preliminary results are obtained as quickly as possible i.e. within a few hours. This is the only way the number of tests to be run can be determined while testing is in progress.
- (b) The number of tests requested above are only estimates. Close liaison with the Task Officer is required to insure that adequate testing is performed and that unnecessary testing is avoided.
- (c) In the event probe or tunnel losses are found, system modification and additional testing will be done as agreed upon at that time between the contractor and the task officer.



13. Provide additional equipment to complete sampling system including rotometer, dry gas meter, filter, sampling pump, and alarm system to indicate loss of critical flow.
  14. Provide mobile cabinet to house above components and install components to provide complete working system.
  15. Calibrate dry gas meter and verify that complete system is operational
  16. Provide dilution air filter and heater assembly similar to assembly in use for tunnel validation tests performed during Nov - Dec, 1976.
- \* Items 7 and 9 were deleted in favor of increased testing on item 12(c).

## APPENDIX B

VELOCITY TRAVERSE OF 21 cm DIAMETER TUNNEL

BY M.N.I.

PC-4

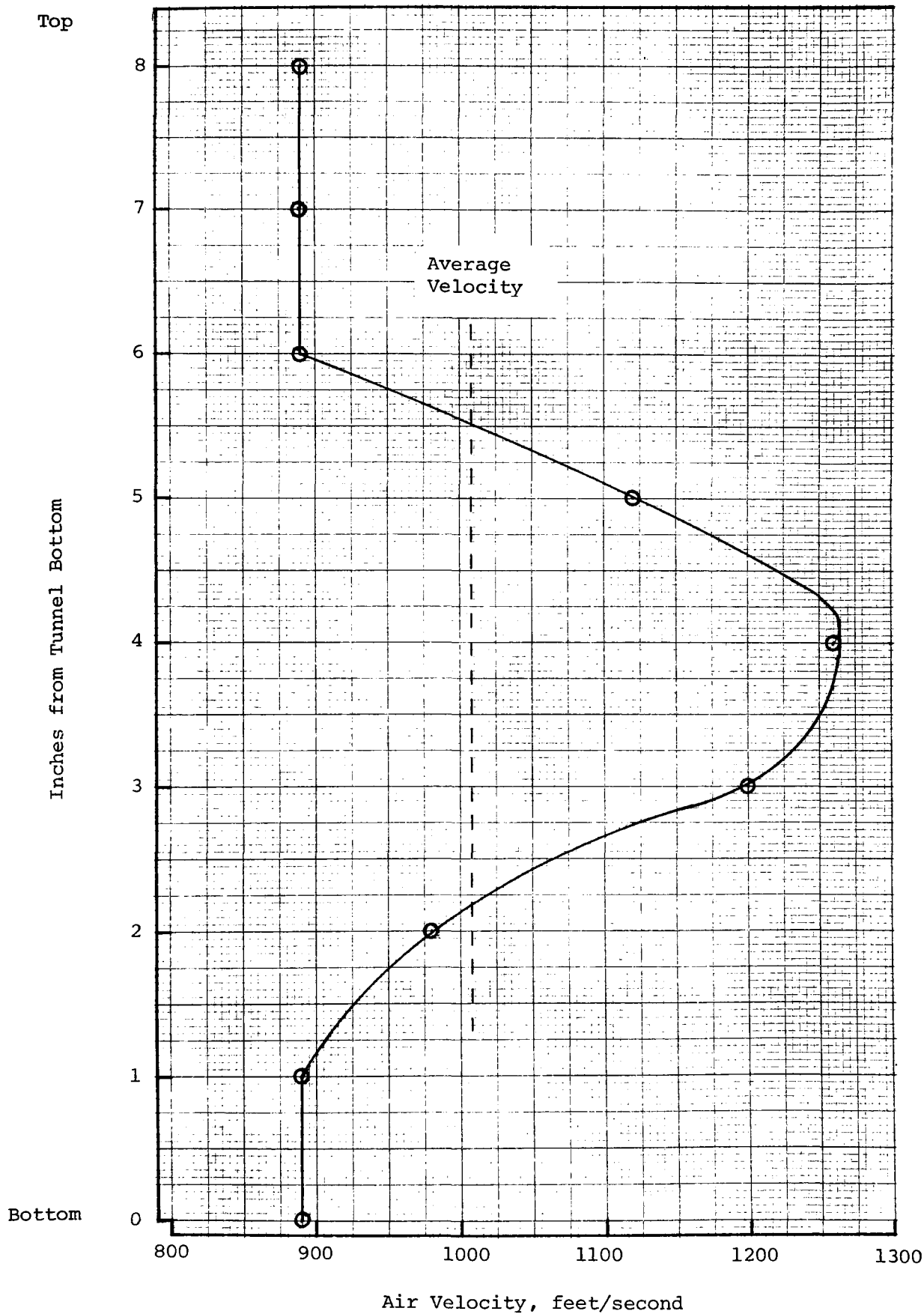


FIGURE B-1. TUNNEL VELOCITY PROFILE

Comparison of Measured Average Velocity  
and Average Velocity from Volume Flow Calibration

(Area of Tunnel = 0.385 ft<sup>2</sup>)

1. CVS blower flow at 28.0 in. Hg.A and 70°F = 381.7 ACFM from blower calibration curve
2. Volume flow at tunnel condition (29.1 in. Hg.A and 71°F) during traverse.

$$V_T = 381.7 \times \frac{28.0}{29.1} \times \frac{531}{530} = 368 \text{ ACFM}$$

3. Average velocity =  $\frac{368}{0.385} = 956 \text{ ft/min}$
4. From velocity profile curve intergrated average velocity is 1009 ft/min. This is only 5.4 percent higher than average velocity from volume flow, which is considered good agreement. Part of the difference is undoubtedly because 890 ft/second was taken as the velocity all the way to the wall (were the velocity is in reality zero) since the velocity profile was not obtained close to the wall.
5. Maximum velocity =  $\left(\frac{1265}{1009}\right) \times \text{Average velocity} = 1.25 \times \text{average velocity}$
6. If volume flow average is used, and maximum velocity is taken to be 1.25 times average velocity, then the design velocity for the sulfate probe is 1195 ft/m (956 x 1.25), since the probe is to be in the area of maximum velocity.

## APPENDIX C

### MIST GENERATOR CALIBRATION DATA

# APPENDIX C - MIST GENERATOR CALIBRATION DATA

## Test I - Determination of Mist as a Percent of Total H<sub>2</sub>SO<sub>4</sub> Input

<u>Run</u>	<u>Test Time</u>	<u>Total ml in Generator</u>	<u>Mist Ml</u>	<u>Percent Mist</u>	<u>Air Press (psi)</u>
1	7'0"	25	2	8.0	24
2	6'43"	25	2	8.0	20
3	10'39"	25	3	12.0	40
4	7'57"	25	3	12.0	30
5	7'05"	25	3	12.0	25

## Test II - Optimization of Compressed Air Pressure

<u>Run</u>	<u>Test Time (sec)</u>	<u>Total ml into Generator</u>	<u>Air Press. (psi)</u>
1	140	1 ml	0
2	19.8	"	30
3	17.7	"	20
4	19.0	"	30
5	21.0	"	35
6	24.0	"	40
7	31.0	"	10
8	20.0	"	15
9	20.0	"	16
10	17.0	"	18
11	16.5	"	20
12	16.5	"	22
13	17.0	"	24
14	17.5	"	26