

NBSIR 73-414

Building and Evaluation of a Polluted Air Delivery System

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Fluid Meters Section
Mechanics Division
National Bureau of Standards
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Final Report on PADS 1

Prepared for
**Environmental Protection Agency
National Environmental Research Center
Quality Control Branch
Chemstrand Bldg.
Research Triangle Park, N. Carolina 27711**



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U. S. DEPARTMENT OF COMMERCE, Frederick B. Dent, Secretary
NATIONAL BUREAU OF STANDARDS, Richard W. Roberts, Director

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Building and Evaluation
of a Polluted Air
Delivery System

ABSTRACT

The building and evaluation of a prototype SO₂ polluted air delivery system (PADS) is discussed. The delivery system was built to delivery sulfur dioxide (SO₂) in air at a rate of 5 liters per minute with design concentrations by volume of 1.0, 0.1 and 0.01 parts per million. It consists of a diluent air delivery system utilizing a critical flow sonic nozzle and three separate concentrated SO₂ in air flow systems utilizing laminar flow porous plugs, one plug for each desired output concentration. The delivery system is contained in a dispatch case and the two gases are delivered to it from pressurized containers through detachable supply lines. Prospective use by unskilled technicians dictated simplicity and durability and compactness.

By maintaining specific upstream pressures on the critical flow nozzle and the laminar flow porous plugs of 45 and 12 psig respectively, the prototype PADS produced average output concentrations of 0.76, 0.100 and 0.003 parts per million of SO₂ in air based on concentration measurements with an NBS calibrated analyzer. The expected output concentrations were 0.98, 0.105 and 0.010 respectively, based on flow calibrations of the individual components. The uncertainty of the output concentration is estimated to be about ± 10 percent.



List of Symbols Used

- A, B and C are the valves selecting the flow at C_1 , through the porous plugs for nominal concentration 1.0, 0.1 and 0.01 ppm respectively.
- A is the cross sectional area, in^2
- B is the barometric pressure in psia.
- B_S is the standard barometric pressure, 14.696 psia.
- C is the output concentration of the system, ppm
- C_1 is the known inlet concentration of SO_2 in air supplied to the porous plugs, ppm
- C_d is the discharge coefficient of the nozzle
- C^* is a critical flow factor for real gas effects
- P_1 is the inlet gage pressure for the critical nozzle in psig.
- P_2 is the inlet gage pressure for the porous plugs in psig.
- Δp_2 is the pressure drop existing across the porous plug.
- Δp_3^* is the pressure drop existing across the piping downstream of the porous plug to the discharge, psi, assumed to be zero for the evaluation of this unit.
- $(Q_0)_N$ is the volume flow rate of the critical nozzle at outlet conditions where the density is ρ_0 , $\text{ft}^3\text{sec}^{-1}$
- $(Q_0)_p$ is the volume flow rate of the porous plug, $\text{ft}^3\text{sec}^{-1}$ at outlet conditions, where the density is ρ_0 .
- R is the gas constant, $0.3705 (\text{lb}_f)(\text{lbm}^{-1})\text{ft}^3\text{in}^{-2}\text{°R}^{-1}$, for air
- R^* is a constant = $0.007677 \text{in}^4\text{atm}^2\text{sec}^2\text{lbm}^{-2}\text{°R}^{-1}$ for air.
- S is the estimate of the standard deviation, percent
- T is the absolute temperature of the flowing gas °R
- T_N is the absolute temperature of the inlet gas to the nozzle, °R
- μ is the viscosity of the gas flow at C_1 , $\text{lbm ft}^{-1}\text{sec}^{-1}$
- ρ_0 is the density at the outlet conditions, lbm ft^{-3}

* Inadvertant or intentional changes of the plumbing downstream from the porous plugs would require a non-zero value of Δp_3 to be used.



Polluted Air Delivery System

1. Introduction

This report covers the design, building and evaluation of a prototype polluted air delivery system, PADS, for use in calibration of atmospheric pollutant analyzers. The PADS produces known concentrations of sulfur dioxide, SO₂, in air streams to be used in the field by unskilled personnel to check the performance of SO₂ analyzers. This program was established by the Environmental Protection Agency at the National Bureau of Standards in order to apply its capabilities to the needed research, development, evaluation and calibration of a gas delivery and calibration system for quality assurance of EPA air programs. The output of this program will be used in quality assurance activities, specifically interlaboratory performance evaluations.

2. The Polluted Air Delivery System

The PADS was designed to produce SO₂ pollutant concentrations of 0.01, 0.1 and 1.0 parts per million by volume (ppm) in air. Mixtures of these concentrations were produced by mixing air containing 2000 ppm of SO₂ with pure air flowing at a rate of 5 liters per minute. The 2000 ppm mixture was selected because the flow of pure SO₂ would be too small to be conveniently and accurately metered. A less concentrated mixture would be more convenient; however, mixtures containing much less than 2000 ppm are reportedly not stable for as long a period of time as the 2000 ppm mixture.

The three dilute mixtures are produced as follows. The diluent air flow of actually 5.7 liters per minute, used for all three mixtures, was metered with a critical flow nozzle having a throat diameter of 0.0156 inches and operated with an upstream pressure, P₁, of 45 psig. The 2000 ppm pollutant, actually 2230 ppm, was metered through one of three porous plug laminar flow restrictors at a time, operated at an upstream pressure of 12 psig. These flows are nominally 0.025, 0.25 and 2.5 cc/min for the nominal 0.01, 0.1 and 1.0 ppm concentrations respectively.

A schematic diagram of the PADS is shown in figure 1. This specific design was chosen to satisfy the EPA's controlling considerations. Namely that the unit use conventional off the shelf items, be small and light, simple to set up and operate, have a minimum of dials to be read and reported or things to be set, be inexpensive, and have an output uncertainty of less than 25 percent of concentration. The critical nozzle was chosen because it is a metering device in which only the upstream temperature T_N and absolute pressure (P₁+B), which must be greater than twice the value of the barometric pressure, need to be known to accurately determine the flow rate through it. The porous plug laminar flow restrictor was chosen to meter the low flows of the concentrated pollutant because of its low flow characteristics and easy replacement. For accurate flow determination the midpoint

temperature and absolute pressure $P_2 + B - \Delta p_2 / 2$ are needed as well as the differential pressure across the device. Both these flow devices will meter flow with an uncertainty of less than 1/2 percent when sufficient care is taken in making these measurements. However this degree of accuracy is not needed in this application. Since that is the case and a minimum number of items to be recorded is desired, it was decided to assume an atmospheric discharge pressure B for both devices and only measure the upstream static pressures, P_1 and P_2 . Considering total cost, size and ruggedness, gages with 1 percent full scale uncertainty were chosen to measure these pressures.

The critical nozzle was made at NBS and the porous plug restrictors were purchased from a vendor. With the exception of the outlet manifold, figures 4, 5 and 7, the entire flow system was constructed of stainless steel parts. The manifold block was of mild steel and the two outlet nozzles were chrome plated brass. The insert, seen in figure 7, in the analyzer supply nozzle of 1/4 inch O.D. stainless steel tubing was added in an effort to help stabilize the output concentration. Bellows-sealed stainless steel valves were used in the system to eliminate the possibility of leakage that might occur in a valve with stem packing. The input connections for the pollutant and pure air to the apparatus are of different sizes to eliminate the possibility of interchanging these connections. A dispatch case was chosen to house the system so that it would be small, easily carried and shipped. All piping, valves, etc. were imbedded and fastened between styrofoam to secure the system in the case and to provide shock absorption.

The PADS was evaluated by measuring the concentration C of the output gas with an analyzer for SO_2 calibrated by the Air Pollution Analysis Section of NBS. See the appendix, page 19 and 20, for the analyzer calibration data. The analyzer was calibrated using its analog output voltage and not the visual dial readings. The analog output of the calibrated analyzer was read with a calibrated recording voltmeter. The visual dial readings were up to 20 percent higher than the analog output and were not used for evaluation of the PADS.

3. Evaluation of the PADS

3.1 Requirements. The experiments performed in the evaluation of the PADS were designed to provide the necessary information to determine the following PADS characteristics as mutually agreed upon by EPA and NBS. The various desired performance characteristics are listed below:

1. Kinetic behavior of output concentration for each restrictor.
2. Effect of order of pollutant flow restrictor use on behavior of output concentration.

3. Effect of ambient temperature (60 to 90°F) on output concentration and output kinetics.
4. Reproducibility of output concentration.
5. Noise in steady state.
6. Effect of poor packing and shipping on output concentration, noise and reproducibility.
7. Effect of fraction bled off on output concentration, noise and reproducibility.
8. Sensitivity of output concentration to gage settings.
9. Estimate of ease of repeating a given gage setting.
10. Estimate of total time from opening of shipping box to full operation based on experience of novice.

3.2 Derived System Performance. An evaluation of the experimental data can be assisted by a development of expected performance as based on flow equations for the meters involved. As stated before, two meters are used in the PADS. Mass flowrate in a critical nozzle when operated over a narrow range of conditions is practically proportional to the inlet absolute pressure divided by the square root of the temperature. Volume rate of flow through a porous plug can be based on Poiseuille's law for incompressible flow through a capillary tube modified for gas compressibility. As derived in the appendix, equation H, the output concentration C can be expressed by the relation

$$C = C_1 \frac{(Q_0)_p}{(Q_0)_N} = C_1 \left[\frac{(P_2 - \Delta p_3) \left(1 + \frac{P_2}{2B} + \frac{\Delta p_3}{2B} \right)}{P_1 + B} \right] \frac{B (\text{constant})}{\mu \sqrt{T}} \quad (1)$$

The constant in (1) should depend only on the portion of the system used as selected by valves A, B or C. Evaluation of the constant can be approached (a) through measurement of C and the various physical variables in the right side of (1), or (b) through measurement of the ratio $(Q_0)_p / (Q_0)_N$ as in volume rate provers and the variables on the right side of (1). C_1 is the concentration of the inlet SO₂ mixture, μ its viscosity and Δp_3 the pressure drop of the downstream piping from the valves to the outlet. Δp_3 is assumed to be zero for the evaluation of this unit.

3.3 Experimental Results. The majority of the evaluation tests were performed in a laboratory where the temperature ranged between 23 and 26°C. The barometer varied between 75 and 76 centimeters of mercury. The SO₂ in air, C, and the pure air were purchased from a vendor and the SO₂ was analyzed by the Air Pollution Analysis

section of NBS to be 2230 ± 22 parts per million. The pure air was checked with the calibrated analyzer for background sulfur dioxide content which was found to be effectively zero. The expected output concentrations were calculated from the relation of the critical nozzle and the porous plug flows, as follows:

$$C = C_1 \frac{(Q_0)_p}{(Q_0)_N} \quad (2)$$

The volume flow through the porous plug and the volume flow through the critical nozzle, were determined by calibration with small positive displacement provers. The gages measuring the pressure on the flow elements were calibrated on an air lubricated dead weight tester. The gage calibrations are in the appendix, page 16. In order to assess many of the performance characteristics of the PADS, a set of tests was designed whereby various flow pressures at the nozzle and pollutant flow restrictor were chosen and random combinations of the two required pressures were used to obtain output concentrations, table 2. The results are discussed and numbered below in the same order as previously listed for the desired performance characteristics. The results are also listed in table 1 and comparison plotted in figure 10. Because of the lack of calibration, resolution and accuracy of the analyzer at and below 0.01 ppm, the results at this concentration will not be discussed but are tabulated and plotted for interest.

1. It was agreed, between EPA and NBS, that the magnitude and repeatability of the time periods to reach 90 and 95 percent of the equilibrated output concentration, as determined from the recorder tracings, would satisfy the question of kinetic behavior of the output concentration. Even though the final equilibration time periods occasionally were as long as one hour, especially for the 0.01 ppm concentration, the periods required to attain 95 percent of the final output concentration value were 10 minutes or less for nominal concentrations of 1.0 and 0.1 ppm. To obtain one test analysis for the SO_2 at each of the three nominal design concentrations, at the 95 percent level of equilibration time, requires less than the two hour maximum time period as desired by EPA.
2. There didn't appear to be any effect due to the order in which the pollutant flow restrictor selector valves were used, but if the pollutant flow is started before the diluent air is turned on the equilibration time increases significantly.
3. To determine the effect of ambient temperature on the PADS, it was moved to a room where its temperature could be maintained at 60 and 90°F. There was no change in output concentration with the 30° temperature change at the nominal 1.0 ppm concentration

and about a 14 percent increase in output concentration with a 30°F increase in temperature at the nominal 0.1 ppm level. A calculation of the expected output concentration change due to a 30° increase in operating temperature using equation (J) in the appendix, indicates a decrease in output concentration of approximately 7 percent should be expected for any one of the desired outputs. The possible increase in output concentration due to the temperature increase is unexplained. Possibly the higher temperature caused desorption of the SO₂ that had saturated the inner wall surfaces. These observations were performed over a period of 4 hours, possibly insufficient to reach equilibrium.

4. The reproducibility of the output concentration was also obtained from the characterization test sets as well as other data at the design conditions. Equation (1) is derived to describe the system performance. A test of its validity can be made by inserting experimental values from Table 2, using corrected values of P₁ and P₂, to determine if the quantity

$$\left(\frac{C_1}{C}\right) \frac{(P_2 - \Delta p_3) \left(1 + \frac{P_2}{2B} + \frac{\Delta p_3}{2B}\right)}{(P_1 + B)} = K \quad (3)$$

is a constant for these tests in which B, T and μ were essentially constant. For valves A, B and C, the quantity K had average values of 803, 6121 and 202,470 respectively, with estimates of standard deviation S of 4.4, 4.6 and 61 percent respectively. The relatively small values of S for valve A and valve B indicate (a) that the equation (1) is valid and (b) that the analyzer repeatability and accuracy are poor near 0.01 ppm considering the large value of S when valve C is used. These values for the constant K apply only at the conditions which existed during evaluation at NBS. Any other evaluation of the unit at different conditions would need different values of K derived by application of equation (1).

5. Noise in the steady state condition was measured from the width of the recorder trace of the recording voltmeter. An indication of the magnitude of the noise can be seen on the sample recorder output, figure 2, and listed in table 1. Its magnitude is of the order of 2 percent for the nominal concentration of 1.0 and 0.1 ppm.

6. To evaluate possible shipping effects, the PADS was shipped via Air Freight to EPA in North Carolina in a cardboard box with little padding between box and dispatch case. After being inspected there, it was repacked in the same box and returned in the same manner. There was no visible damage to the case or its components upon its return. The PADS was then retested against the analyzer and the output concentration values were found to be within the range of values obtained before shipping. It is hoped that normally the PADS will be packed, possibly with the pressure regulators and

maybe the pollutant bottle, in a re-useable container that satisfies "Air Transport Association" (A.T.A.) specifications number 300, category 1.

7. An attempt was made to determine the effect of increasing the mixture flow to the analyzer. This flow was varied by bleeding off additional mixture through an auxiliary tube, figure 3. This procedure was followed because it was not possible to vary the flow extracted by the analyzer. The additional flows were bled off through a calibrated laminar flow meter at rates of 1.5, 3 and 4.2 liters per minute. As can be seen in figure 10, there was no measureable effect at the nominal 1.0 ppm concentration. The output values were all within the spread of output concentrations without any bleed. The output values, with additional bleed, for the nominal 0.1 ppm concentration were within the previous spread of output values without bleed except at one rate, 1.5 lpm, where the concentration was lower than the previous data.

8. Information on the sensitivity of the output concentration to the setting of the gages can be obtained from the results of the characterization test sets, table 2, as expressed by equation (3), or as expressed by differential equation (J) in the appendix. The effect of pressure change on concentration is -1.7 percent for a one psi increase in P_1 , and 11 percent increase in output concentration for a one psi increase in P_2 . These are listed in table 1 in terms of concentration, ppm/psi for the two highest output concentrations.

9. Repeating a given pressure setting to within 1 percent of the design pressure is not difficult. The pressure adjustment with the regulators should be made while lightly tapping the PADS's gages and the line of sight should be normal to the face of the gage.

10. The last item on the list of characteristics to be investigated is a request for an estimate of the time required for a novice to put the PADS in operation. In our opinion it should not take any longer than one hour.

The evaluation of the PADS was performed with the flow configuration as shown in figure 4. Referring to figure 10, the data at test one was taken with the piping arranged similar to figure 5 but with the porous plugs to the left or downstream of the selector valves. For test number two, figure 10, the valves and plugs were switched, figure 5, to see if the arrangement would decrease the equilibration time. This arrangement was a marginal improvement. For the remainder of the tests the piping was arranged as in figure 4. The downstream piping from the selector valves to the mixing tee was replaced with 0.010 inch inside diameter capillary tubing. The installation of the capillary tubing made a marked improvement in the equilibration times. The porous plugs were left upstream of the selector valves so that the plugs could be kept saturated at the supply pressure of the pollutant.

It could be assumed that changing the position of the valves and plugs, and the addition of the tubing, caused the decrease in output concentration, shown in figure 10, by increasing the back pressure on the porous plugs which would have decreased the pollutant flow and thereby the output concentration. A theoretical calculation of the pressure drop, using equation A, indicated that the pressure drop across the downstream capillaries with inside diameters of 0.01 inch would decrease the flow and the concentration 2.5 percent for the largest pollutant flow, .2 percent for the middle flow, and .015 percent for the lowest. These calculated decreases in pollutant flows and concentrations are smaller than the observed concentration decreases of from 10 to 50 percent. A calculation, based on equations 2, A and J, indicated that the diameter of the capillary tubing would have to be .006 and .004 inch rather than the presumed .010 inch to cause the decrease in output concentration exhibited for the nominal 1.0 and 0.1 ppm portions of the PADS system. It is felt that the decrease can not be attributed solely to a smaller than expected capillary, but to unknown effects.

Accuracy of the results should be estimated through comparison of results obtained by two or more independent methods. In this case a second method and its results are available from use of equation 2 and flow calibrations of the porous plugs and critical nozzle as listed in the Appendix, page 17. The output bias is defined as the difference between the average value of the output concentration (as calculated from the average value of K, equation 3) and the concentration calculated from equation 2. This difference is -0.22 and -0.005 ppm, respectively, at the nominal concentrations of 1.0 and 0.1 ppm. An estimate of the overall uncertainty of the flow calibrations of about 3 percent cannot account for the difference of 0.22 ppm which on a percentage basis is about 22 percent. The difference of 0.005 ppm is reconcilable considering the estimated uncertainty of 3 percent for the flow calibrations and about 2 percent for 3 times the standard error of the reported values of the concentration by the analytical procedure. Without further experimentation and intercomparison of methods, and considering both results, it appears that the accuracy cannot be estimated as better than about 10 percent of output concentration.

4. Operating Instructions

The following steps are necessary to install the PADS in the field and make it ready for operation. (Fig 6)

1. Assemble the gas bottles, regulators, connecting tubing and PADS at the site of the analyzer to be checked.
2. Connect the regulators to the gas bottles.
3. Attach the tubing between the regulators and the PADS inlet connections and between the PADS outlet manifold nipples and analyzer and exhaust.

4. Set the diluent air and SO₂ gas flow pressures, 45 and 12 psi respectively, on the PADS gages with the pressure regulators. Take care to lightly tap the PADS gages while setting the pressures and to view the gages normally to their faces. Diluent air will be flowing at this point.
5. Before any data is taken it should be determined that nothing is restricting the flow from the outlet manifold nozzles. Otherwise the output concentrations would be erroneous due to the higher back pressure created by the restriction.
6. Open valve A and allow 30 minutes to achieve an equilibrium state.
7. Read and record the PADS gages gas pressures and the concentration indication of the analyzer being checked.
8. Close valve A and allow about 5 minutes before opening valve B.
9. Repeat steps 6, 7, and 8 for valve B and then C.
10. If available, a barometer reading, and area temperature reading should also be recorded at the same time as the data in step 7.
11. In order to compare the output concentrations of the PADS, as indicated by field analyzers, with the calibrated output concentration of the PADS, it is necessary to make corrections to the calibration output concentration, for the conditions that existed at the field analyzer. This can be done by using the corrections in equation J of the appendix in the following manner. Let a "o" subscript designate the output concentration at the time of PADS calibration and "n" designate the output concentration at the time of comparison with a field analyzer. C_c will designate the PADS calibration output concentration corrected to field analyzer conditions.

$$C_c = C_o \frac{C_{1n}}{C_{1o}} \left(1 + \frac{1.3(P_{2n}-P_{2o})}{P_{2n}} + .46 \left(\frac{B_n - B_o}{B_n} \right) - 1.3 \frac{T_n - T_o}{T_n} - .75 \frac{P_{1n} - P_{1o}}{P_{1n}} \right)$$

The differential change of C₁, inlet concentration of SO₂, is not used above in the event that C₁ varies widely from one supply bottle to the next. As stated in item 3 of 3.3, the measured effect of temperature change on the output concentration is not fully understood and does not agree with that in the above equation.

12. During routine inspection of the PADS, between its uses in the field, the PADS should be recompared to a calibrated analyzer. During this test the pressure set on the SO₂ regulator pressure gage, to attain the necessary pressure on the PADS SO₂ gage,

should be recorded. If and when in subsequent inspections an increase in the difference between the two pressures is noted it is likely that the filter element needs to be replaced, barring any leaks in the connections between the regulator and filter.

13. Inspection for leaks from the PADS can be accomplished by removing the system from the case and using a commercial leak detection liquid, or a soap and water solution, on the connections while the system is pressurized to its normal pressures.

5. PADS Specifications

1. All parts that would come in contact with the SO₂ mixture were purchased in stainless steel.
2. All parts that would come in contact with SO₂ except the gauges, valves, porous plugs, filters, quick disconnects and capillary tubing, were cleaned in 30 percent nitric acid solution and rinsed in distilled water.
3. The valves are of the bellows sealed type, installed so that the bellows is on the upstream side of the valve or the higher pressure side of the valve.
4. The gages have stainless steel bourdon tubes and pressure scales of 15 and 60 psi respectively. They have manufacturers stated accuracies of 1 percent full scale and were calibrated at NBS on an air lubricated dead weight tester.
5. The stainless steel porous plugs which restrict the pollutant flow are assembled into 1 3/8 inch long, 1/4 inch O.D. stainless steel tubing. They were ordered with nominal flowrates of 0.025, 0.25 and 2.5 cc/min when the differential pressure across them is 12 psig with atmospheric discharge.
6. The critical flow nozzle, figure 8, was made from 1/4 inch O.D. stainless steel rod 1 3/8 inch long. It was drilled out to within 1/32 inch from the opposite end. The nozzle throat was drilled 1/64 inch I.D. in the blank end. The nozzle was shaped by hand. The flow through the nozzle was designed for a nominal 5 liters per minute of air with an upstream pressure of 45 psig and atmospheric discharge, but calibration proved it to be 5.7 liters per minute.
7. All the tube fittings used in the PADS use compressed ferrules as the method of sealing the tubing to the fittings, and fit 1/4 inch O.D. stainless steel tubing.

8. The tubing used between the regulators and the PADS was 1/4 inch O.D. stainless steel for the SO₂ mixture and 1/4 inch O.D., 1/16 inch wall plastic tubing for the air.
9. The regulator used for the SO₂ concentrated pollutant was all stainless steel, sized for the flow range designed for, .025 to 2.5 cc/min. The regulator used for the air was conventional 2 stage air regulator.
10. The assembled flow system of the PADS was sandwiched between styrofoam for shock absorption and fitted into a dispatch case.
11. After final assembly the PADS was evaluated against a calibrated SO₂ analyzer at normal room temperatures and barometric pressure, and at room temperatures of 60° and 90°F.

6. Recommendations

1. It is felt that stainless steel (316) or Teflon should be used wherever SO₂ will be in the system.
2. All stainless steel parts of the system, except those which have other components that would be damaged, should be cleaned in a 97 percent nitric acid and 3 percent hydroflouric acid solution for 10 minutes at room temperature, rinsed in distilled water and dried.
3. The valves should be of the bellows sealed type, constructed of 316 stainless steel and installed with the bellows on the upstream side of the valve.
4. The gages should have 316 stainless steel bourdon tubes and be accurate to within 1 percent of full scale. The pollutant gage should be 15 psi full scale and the pure air gage 60 psi full scale.
5. The porous plug flow restrictors should be made of 316 stainless steel mounted into 1 3/8 inch long 1/4 inch O.D. 316 stainless steel tubing. They should have output flows of 0.025, 0.25, and 2.5 cc/min, when the differential pressure across them is 12 psi and the discharge is at atmospheric pressure, for nominal output concentrations of 0.01, 0.1 and 1.0 parts per million.
6. The critical flow nozzle should be made of 316 stainless steel as in figure 8. A mandrel should be made to shape the inlet radius of curvature. The nozzle could also be made by the electro-forming process and fastened to the end of a 1 3/8 inch long, 1/4 inch O.D. piece of stainless steel tubing.

7. The gages should be checked against a transfer standard to verify their accuracy.
8. The tubing delivering the SO₂ to the PADS from the pressure regulator should be either stainless steel or Teflon. Stainless steel may be more awkward to use because of its rigidity.
9. The completely assembled PADS should be evaluated with a calibrated SO₂ analyzer. The evaluation should include data taken at various pressure levels in order to determine the effect of changing the inlet air and SO₂ pressure on the output concentration. If possible, the evaluation should include data taken at various surrounding temperatures and barometric pressures. Where this is not possible a theoretical correction must be made for data taken at temperatures and barometric pressures different than those taken at the time of initial evaluation, as outlined under "Operating Instructions".

Parts List**

Case - dispatch - NBS storeroom FSC 8460-782-6726*

Critical nozzle - NBS shop made - 0.0156 inch ID, 1/4" OD

Porous plugs - Purchased from Mott Metallurgical Corp.: 2.5 cc/min, 0.25 cc/min, 0.025 cc/min; 0.025 cc/min plug replaced with 0.10 cc/min before unit was returned to EPA.

Gages - Purchased from Marsh Instrument Co.
Part No. 1-BJC-03160-ADL-15 psi
Part No. W 0372 60 PSI

Valves - Hoke 4100 series, Part no. 4172 G4Y

Filter - Hoke series 6300, Part no. 6321G4Y

Quick Disconnects - TOMCO Part no. 3452-R6 and 3472-R2, 1/4" SS.: Part no. 3650-R6 and 3670-R2, 3/8" brass

Capillary tubing - Small Parts Inc. Part no. CTX-6210 .010 inch I.D. 316 S.S.

All tube tees - Hoke part no. 4TTT - S, 1/4" S.S.

Female elbows - Hoke part no. 4LF4-S, 1/4" S.S.

Male connector - Hoke part 4CM4-S, 1/4" S.S.

Tubing - 1/4" S.S. NBS storeroom FSC 4710-914-5137*

Capillary tube end fittings - NBS shop made from S.S. rod FSC 9510-950-3671*

Outlet manifold block - NBS shop made of mild steel

Outlet nozzles - From surplus lab sinks.

* General Services Administration Federal Stock Control number.

** In no case does the identification of the manufacturers in the above listing imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that the item identified is necessarily the best available for the purpose.

Appendix

In order to determine the effect various physical variables have on the output concentration of the PADS, it is necessary to look at the equations of flow of the critical flow nozzle and the laminar flow porous plug.

Laminar flow of an incompressible fluid through a capillary tube is given by the well known Poiseuille relationship

$$Q = \frac{\Delta p \pi D^4}{128 \mu L} \quad (A)$$

Therefore the volume flow is proportional to the pressure drop Δp through the porous plug and the "effective" inside diameter D of the plug, to the 4th power, and inversely proportional to the "effective" length L of the plug and the viscosity μ of the flowing fluid.

When a compressible fluid is used, the results for a laminar device are more consistent and linear if the volume rate is defined at the conditions presumed to exist at the midpoint of the laminar device. P_2 , as measured on the PADS, is the pressure drop across the porous plug laminar flow restrictor and all piping downstream from the plug to the outlet manifold. P_2 can be said to consist of two parts, Δp_2 the drop across the porous plug, and Δp_3 the drop across the piping downstream of the porous plug to the discharge, and

$$P_2 = \Delta p_2 + \Delta p_3. \quad (B)$$

Δp_3 is assumed to be zero for the evaluation of this unit. The midpoint absolute pressure in the porous plugs is given by

$$P'_{2m} = P_2 + B - \frac{\Delta p_2}{2} \quad (C)$$

where B is the barometric pressure. Rearranging (B) and combining with (C) produces:

$$P'_{2m} = B + \frac{P_2}{2} + \frac{\Delta p_3}{2}. \quad (D)$$

Let $(Q_0)_p$ equal the flow rate at outlet conditions where the density is ρ_0 , and the pressure is B . If we assume that the temperature at the outlet of the porous plug is equal to the temperature at the midpoint of the porous plug the relationship between $(Q_0)_p$ and the volume rate Q_p defined at the midpoint pressure in the plug is given by

$$(Q_0)_p = \left(\frac{B + \frac{P_2}{2} + \frac{\Delta p_3}{2}}{B} \right) Q_p. \quad (E)$$

Upon substitution of Q_p for Q and Δp_2 for Δp in equation (A), it becomes

$$(Q_o)_p = \frac{144(P_2 - \Delta p_3) \pi D^4}{128\mu L} \left(1 + \frac{P_2}{2B} + \frac{\Delta p_3}{2B}\right) \quad (F)$$

where the factor 144 converts $(P_2 - \Delta p_3)$ in psi to psf.

For the critical nozzle the mass flow M_N in lbm sec^{-1} is represented by the equation

$$M_N = C_d AC^* \left(\frac{P_1+B}{B_s}\right) / \sqrt{R^*T_N} \quad .$$

If we let $(Q_o)_N$ equal the volume flow rate at the outlet condition where the density is ρ_o in lbm ft^{-3} , then

$$(Q_o)_N = \frac{C_d AC^*}{\rho_o} (P_1+B) / B_s \sqrt{R^*T_N} \quad . \quad (G)$$

Previously it was stated in equation (2) that $C = C_1(Q_o)_p / (Q_o)_N$ and inserting equation F and G into it produces:

$$C = C_1 \frac{(Q_o)_p}{(Q_o)_N} = C_1 \left[\frac{(P_2 - \Delta p_3) \left(1 + \frac{P_2}{2B} + \frac{\Delta p_3}{2B}\right)}{(P_1 + B) / B_s} \right] \left[\frac{\pi D^4 (144) B \sqrt{R^*T_N}}{RT_o 128\mu L C_d A C^*} \right] \quad (H)$$

where B/RT_o from the gas law has been used in place of ρ_o .

Equation (H) can be used as is to evaluate effect of changes of the physical variables on C , or its differential can be used for the same purpose. Taking the differential of the log of C produces

$$\begin{aligned} \frac{dC}{C} = & \frac{dC_1}{C_1} + \frac{dP_2}{P_2 - \Delta p_3} \left[1 + \frac{P_2 - \Delta p_3}{P_2 + 2B + \Delta p_3} \right] - \frac{d(\Delta p_3)}{P_2 - \Delta p_3} \left[1 - \frac{P_2 - \Delta p_3}{P_2 + 2B + \Delta p_3} \right] \\ & + \frac{dB}{B} \left[1 - \frac{P_2 + \Delta p_3}{P_2 + 2B + \Delta p_3} - \frac{B}{P_1 + B} \right] - \frac{dP_1}{P_1 + B} - \frac{1.3dT}{T} \\ & - \frac{dL}{L} - \frac{dA}{A} + 4 \frac{dD}{D} - \frac{d(C^*C_d)}{(C^*C_d)} \end{aligned} \quad (I)$$

where du/u is replaced by $0.8 dT/T$, and added to $d(\log \sqrt{T})$, giving $\frac{1.3dT}{T}$.

It can be shown that the sum of the terms $(-\frac{dL}{L} - \frac{dA}{A} + 4\frac{dD}{D})$ is equal to αdT , assuming that the gas temperature for the porous plug and nozzle are the same and, where α is the linear coefficient of expansion for the nozzle and porous plug material. Since stainless steel is used in the PADS, αdT is very small for ordinary temperature variations, and can be ignored.

In the term $\frac{d(C \cdot C_d)}{C \cdot C_d}$, $C \cdot C_d$ varies very little, less than ± 0.1 percent, within the pressure and temperature range which it would normally encounter, and is also ignored.

Equation (I) can be reduced to;

$$\frac{dC}{C} = \frac{dC_1}{C_1} + 1.3 \frac{dP_2}{P_2} + .46 \frac{dB}{B} - .75 \frac{dP_1}{P_1} - 1.3 \frac{dT}{T} - 0.7 \frac{d(\Delta p_3)}{P_2} \quad (J)$$

for operation near the design pressures and temperature for the PADS. The term containing the effect of change of Δp_3 is retained as a reminder that the output concentration can be changed by inadvertant or intentional changes of plumbing downstream from the porous plugs. Equation (J) is a differential equation and is strictly accurate only in the limit of all differential quantities approaching zero.

As an example of the use of equation (J), consider what effect changes in barometric pressure would have on the output concentration. If the barometer pressure increased by 1 percent, the change in output concentration at 1 ppm would be,

$$dC = 0.46 \frac{dB}{B} C = 0.46 (.01)(1\text{ppm}) = + .0046 \text{ ppm.}$$

Gage Calibrations

Marsh 60 psi gage

Type 11-S

<u>Actual pressure</u> <u>psi</u>	<u>Average**</u> <u>Gage Reading</u>
15.21	14.98
20.21	19.82
25.20	24.95
30.20	30.0
35.20	34.9
40.20	39.82
45.19	44.95
50.19	50.1
55.19	55.28
60.19	60.5*

Marsh 15 psi gage

Type 11-S

<u>Actual pressure</u> <u>psi</u>	<u>Average**</u> <u>Gage Reading</u>
5.22	5.4
10.21	10.45
15.21	15.57*

* Estimated overscale readings.
** Averages of 5 readings

Calibration of Porous Plugs*

<u>Nominal Flow Rate cc/min</u>	<u>Porous Plug Pressure psi</u>	<u>Calibrator Volume-cc</u>	<u>Average Calibrator Time-sec</u>	<u>Average Calibrator Flow Rate-cc/min</u>
0.025	11.71	0.1044	251.6	0.025
0.25	11.71	0.2090	45.86	0.27
2.5	11.71	1.2789	30.42	2.52

Calibration of Critical Nozzle*

Nominal Flow 5 liters/min.

Calibrator volume = 1.4999 liters

<u>Nozzle Temp. °C</u>	<u>Calib. Temp. °C</u>	<u>Calib. Press. psig</u>	<u>Barom. psia</u>	<u>Nozzle Press. psig</u>	<u>Calib. Time-sec</u>	<u>Calib. Flow-Lpm @14.7 psia & 20° C</u>
21.8	21.95	.059	14.42	42.65	16.091	5.475
21.8	21.85	.059	14.41	39.65	16.961	5.195
21.85	21.95	.059	14.41	37.65	17.578	5.011
22.05	22.25	.059	14.41	35.65	18.274	4.815
22.35	22.05	.059	14.61	34.65	18.848	4.736
22.35	22.05	.059	14.61	29.88	21.027	4.246

* Five determinations of flow were made at each rate.

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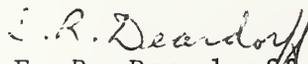
REPORT OF ANALYSIS

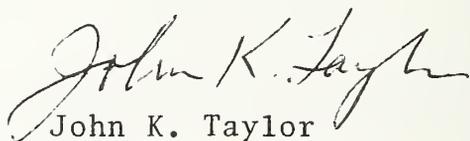
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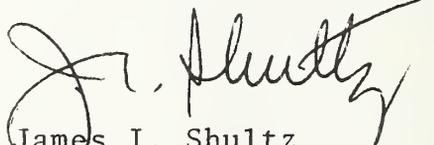
Tank of 0.2 Percent SO₂

Submitted by G. P. Baumgarten, 213.06
Project No. 2130161 Div. Req. No. 213-6003-74

Titration with standardized sodium hydroxide shows 2230 ppm
SO₂ accurate within \pm 22 ppm.


E. R. Deardorff
Chemist


John K. Taylor
Chief
Air Pollution Analysis Section


James I. Shultz
Service Analysis Coordinator
Analytical Chemistry Division

August 1, 1973

74-11a

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REPORT OF ANALYSIS

Calibration of Total Sulfur Analyzer

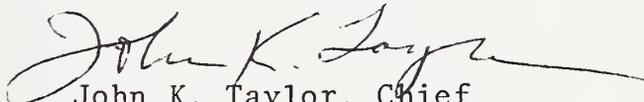
Submitted by G. P. Baumgarten, 213.06
Project No. 2130161, Div. Req. No. 213-6003-74

Two total sulfur analyzers, a Meloy SA-185-2 and a Bendix 8300, were calibrated with air containing known amounts of sulfur dioxide. The concentration of sulfur dioxide was fixed using permeation tubes (NBS Standard Reference Materials 1625 and 1626) and measured flows of air.

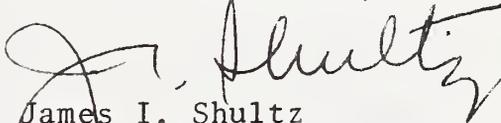
The instruments were calibrated by the procedure specified by the manufacturers. The attached table summarizes the analytical results.



James Wing, Chemist



John K. Taylor, Chief
Air Pollution Analysis Section



James I. Shultz
Service Analysis Coordinator
Analytical Chemistry Division

September 24, 1973

Enclosure

74-11

MELOY SA-185-2***

BENDIX 8300

Concentration of Sulfur Dioxide
in Parts Per Million

<u>Calculated*</u>	<u>Observed**</u>	<u>Calculated*</u>	<u>Observed**</u>
0.953	0.954	0.953	0.941, 0.947, 0.937, 0.935, 0.948
0.795	0.805, 0.810	0.795	0.803, 0.798, 0.798 0.800, 0.801
0.683	0.687, 0.696	0.683	0.695, 0.694, 0.688, 0.686, 0.688
0.533	0.540, 0.550	0.533	0.543, 0.545, 0.540, 0.536, 0.536
0.475	0.492, 0.498		
0.437	0.441, 0.446	0.437	0.444, 0.443, 0.438, 0.434
0.370	0.372, 0.374	0.370	0.372, 0.373, 0.372, 0.370, 0.365
0.324	0.321, 0.328		
0.269	0.264	0.269	0.262, 0.263, 0.262, 0.255
0.198	0.186, 0.188		
0.112	0.098, 0.096		
0.0775	0.065, 0.063		
0.067	0.056, 0.054		
0.056	0.046, 0.046		

* The calculated value is based on the known rate of the permeation tube and the measured flow of air. Estimated inaccuracies in the rates of permeation and of air flow are ± 0.5 percent and ± 2 percent, respectively.

** The observed values were recorded from the analyzers analog output on a calibrated recording volt meter.

*** Used in the evaluation of the PADS

Note: The Bendix unit was borrowed from EPA but not used for this evaluation. It will be used in subsequent work.

Table 1

Conclusions of Concentration Characterization Test Sets

Nominal Design Concentrations

Test factors	Nominal Design Concentrations		
	Valve A 1.0 ppm	Valve B 0.1 ppm	Valve C 0.01 ppm
1. Kinetic behavior { P ₁ = 45 psi P ₂ = 12 psi	2 min ± 1	3 min ± 2	17 min ± 15
2. Effect of Order of Restrictor use :	4 min ± 1	6 min ± 4	25 min ± 20
3. Effect of Ambient Temperature change : from 60° to 90°F	Concentration values within the range of values at 72°F. Average value between 0.76 and 0.79 ppm.	≈14% increase in concentration. 60° values < 72° values 90° values ≈ 72° values 90° Eq.time < 72° Eq.time	≈200% increase in concentration. 60° values ≈ 72° values 90° values > 72° values No change in Eq.time
4. Reproducibility of the average output concentration, S: Average output concentration Expected output concentration	+4.4% .76 ppm .98 ppm	+4.6% .100 ppm .105 ppm	+61% .003 ppm .010 ppm
5. Noise in steady state:	.02 ppm	.002 ppm	.001 ppm
6. Effect of shipping:	No observable differences in any of the output concentrations.		
7. Effect of fraction of output flow bled off:	None	None	Output concentration decreased slightly
8. Sensitivity of output concentration to gage settings: ppm/psi Nozzle pressure, P ₁ : Porous plug pressure, P ₂ :	- .013 + .084	- .0017 + .011	
9. Ease of repeating given : gage settings	This is not difficult with reasonable care, but the gages need to be tapped lightly while setting the pressures.		
10. Estimate of time necessary to set unit up :	No more than one hour should be needed.		

Table 2

Output Concentrations of the Characterization Test Sets

The tabulated values are the corrected analog output of the SO₂ analyzer, in ppm.

P₁, air pressure, and P₂, pollutant pressure, are as read from the PADS gages, psig, and are uncorrected.

Round 1			Round 2			Round 3					
Valve B			Valve A			Valve C					
P ₂	9	12	15	P ₂	9	12	13	P ₂	11	12	15
P ₁ :				P ₁ :				P ₁ :			
30	.097	.138	.177	40	.580	.81	.872	30	.005	.0065	.008
45	.075	.101	.134	45	.533	.732	.815	45	.0003	.0038	.006
60	.057	.085	.106	60	.446	.610	.670	50	.001	.000	.0038
Valve A			Valve C			Valve B					
P ₂	9	12	13	P ₂	9	12	15	P ₂	9	12	15
P ₁ :				P ₁ :				P ₁ :			
40	.573	.795	.850	30	.0044	.0057	.009	30	.096	.130	.171
45	.535	.753	.784	45	.0003	.002	.0055	45	.073	.100	.128
60	.451	.592	.648	50	.0004	.0004	.0045	60	.057	.080	.104
Valve C			Valve B			Valve A					
P ₂	11	12	15	P ₂	9	12	15	P ₂	9	12	13
P ₁ :				P ₁ :				P ₁ :			
30	.0047	.0068	.009	30	.098	.129	.168	40	.605	.83	.904
45	.001	.0023	.0055	45	.069	.097	.120	45	.564	.806	.856
50	.0002	.002	.0048	60	.054	.076	.099	60	.46	.636	.708

POLLUTED AIR DELIVERY SYSTEM

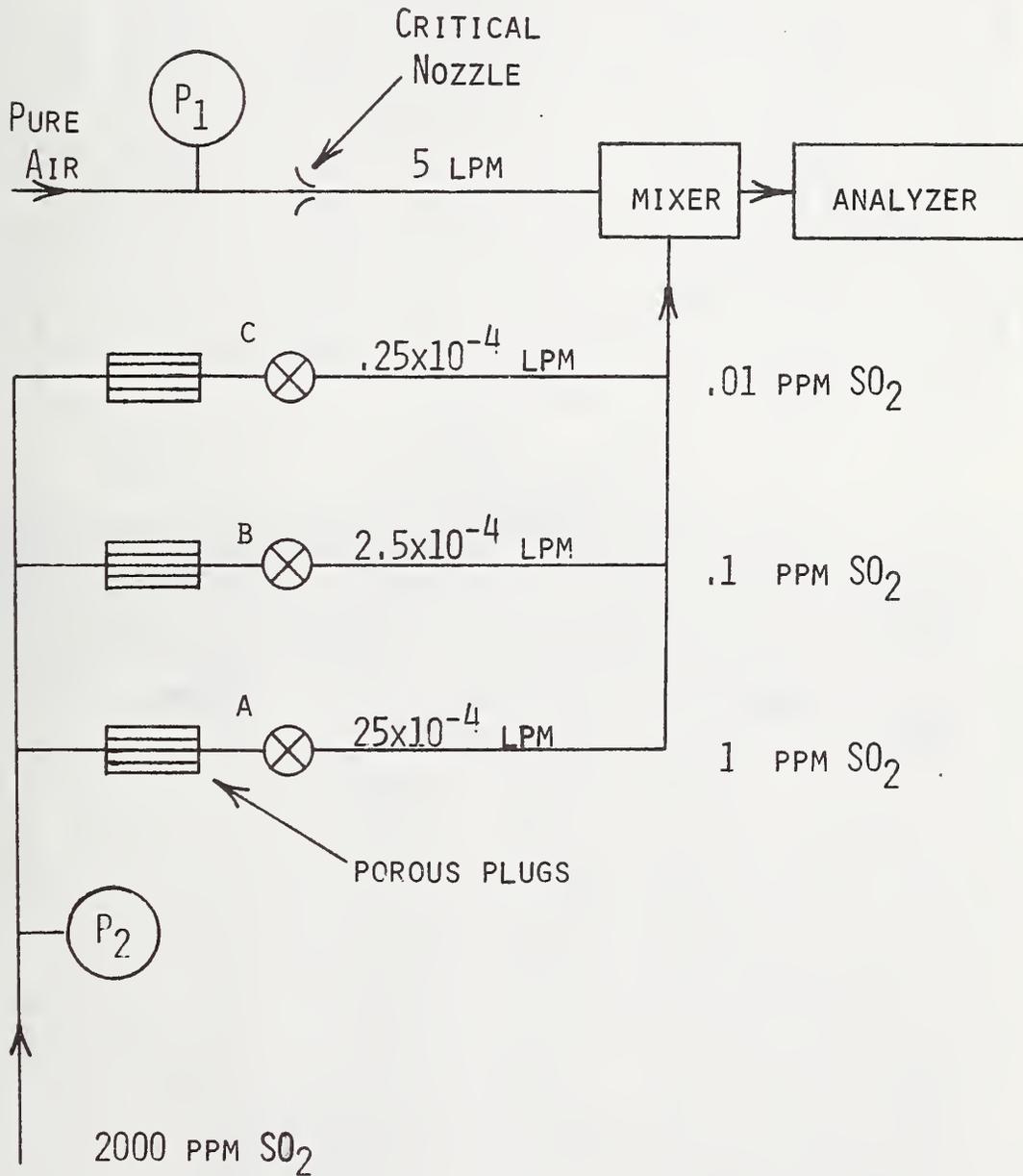
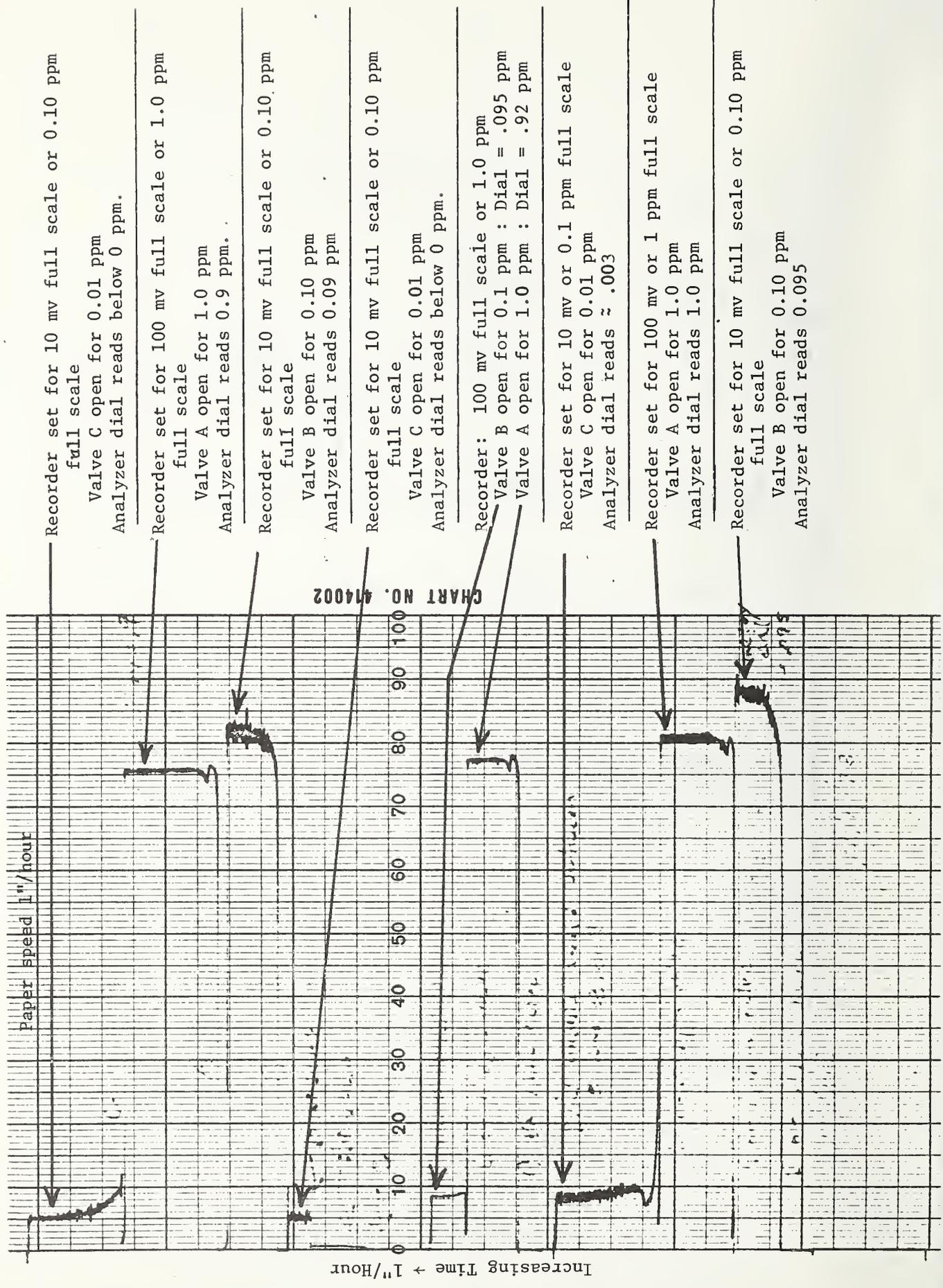


Figure 1

Figure 2
Sample Recorder Output of Analyzer



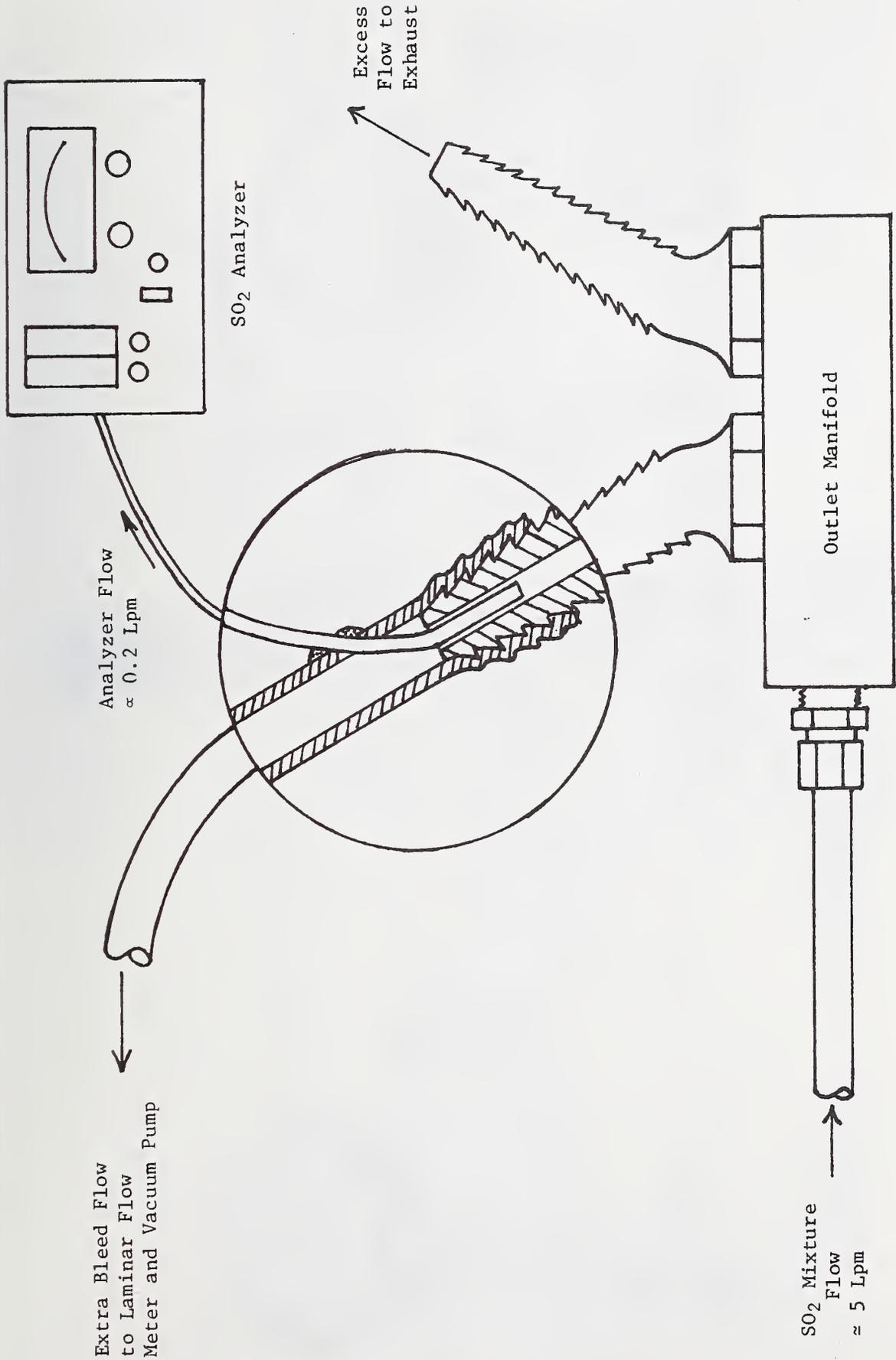


Figure 3 Sketch of Method for Increased Bleed at Analyzer Outlet Nozzle



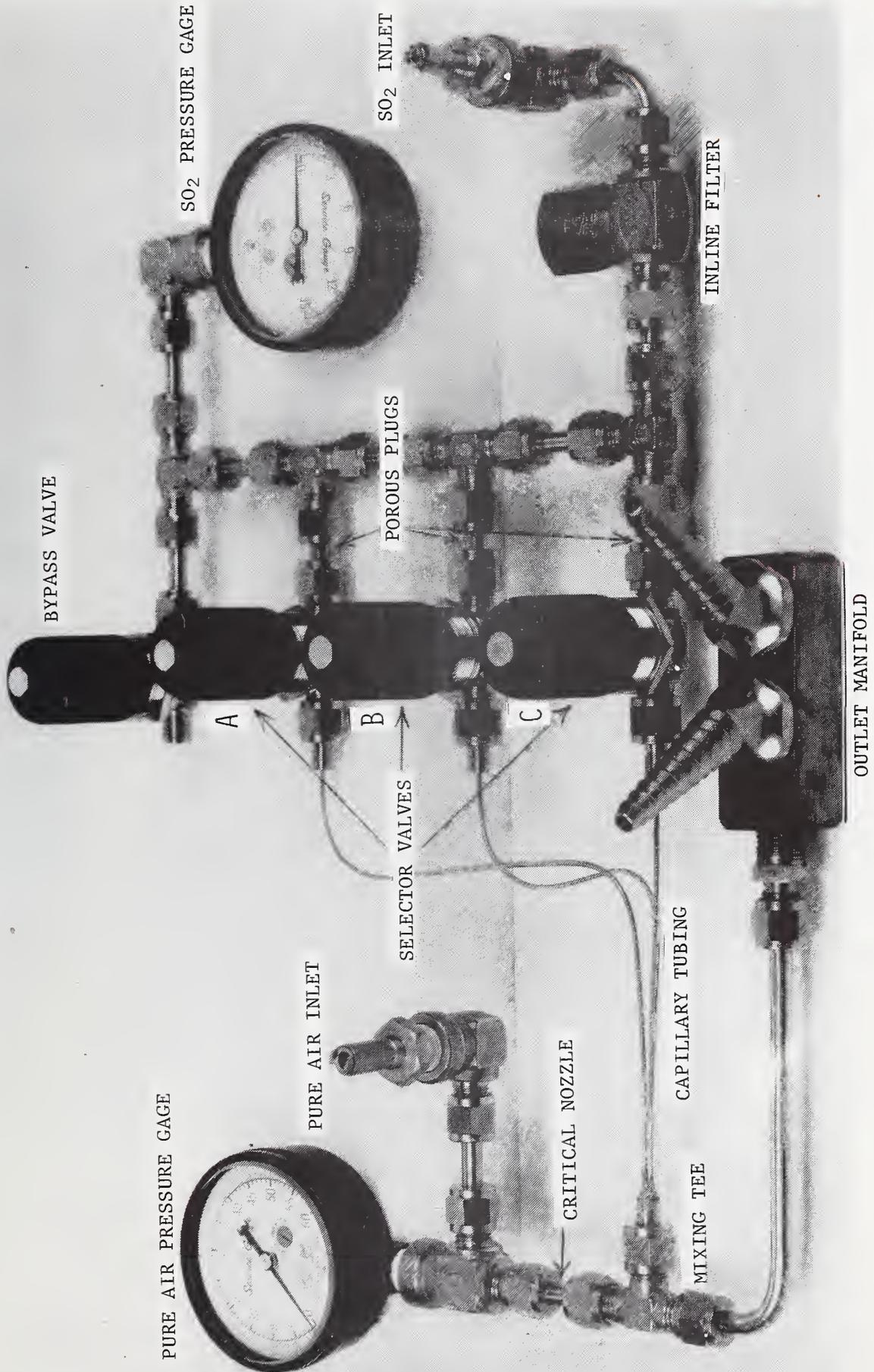


Figure 4



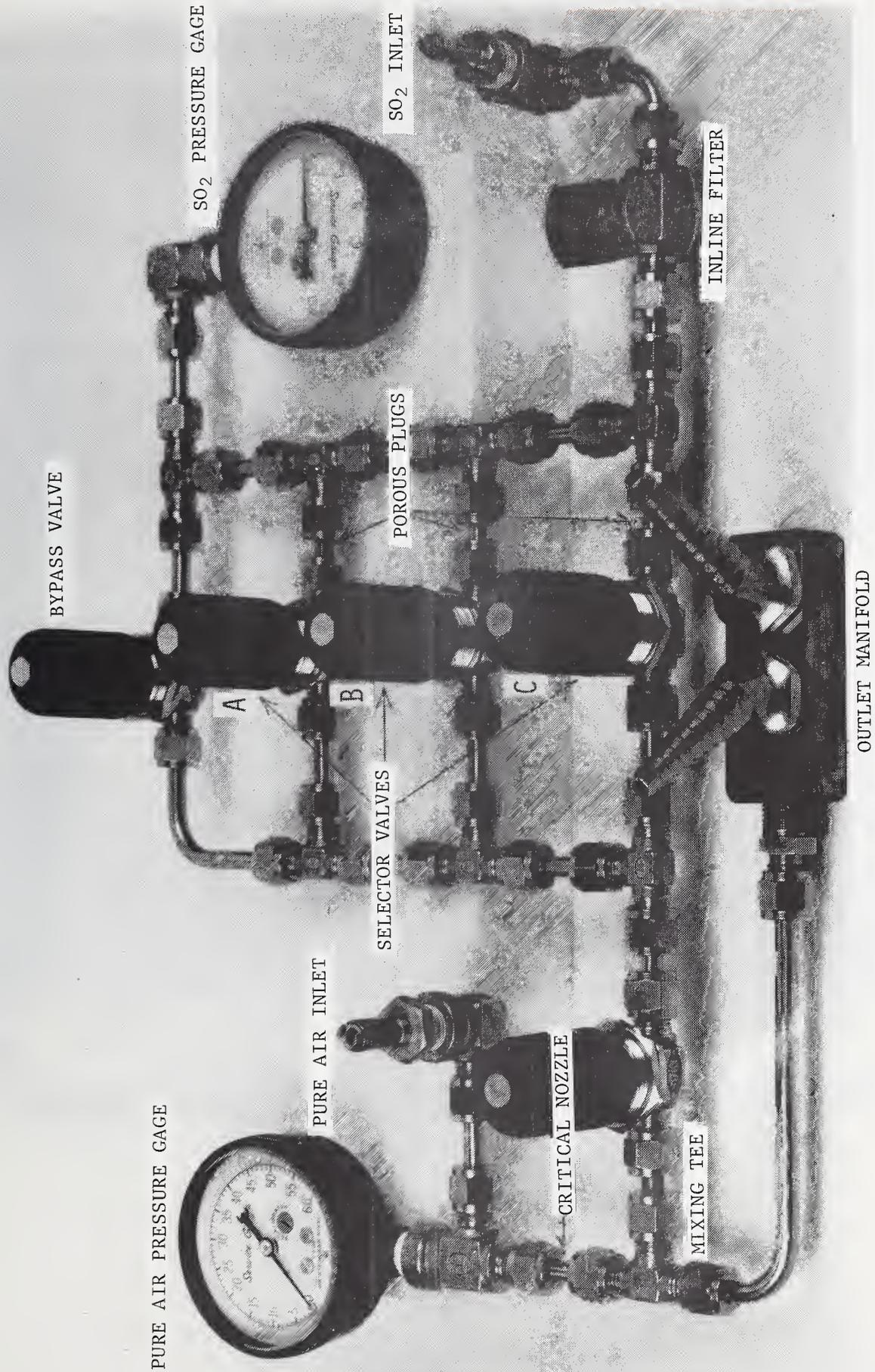


Figure 5



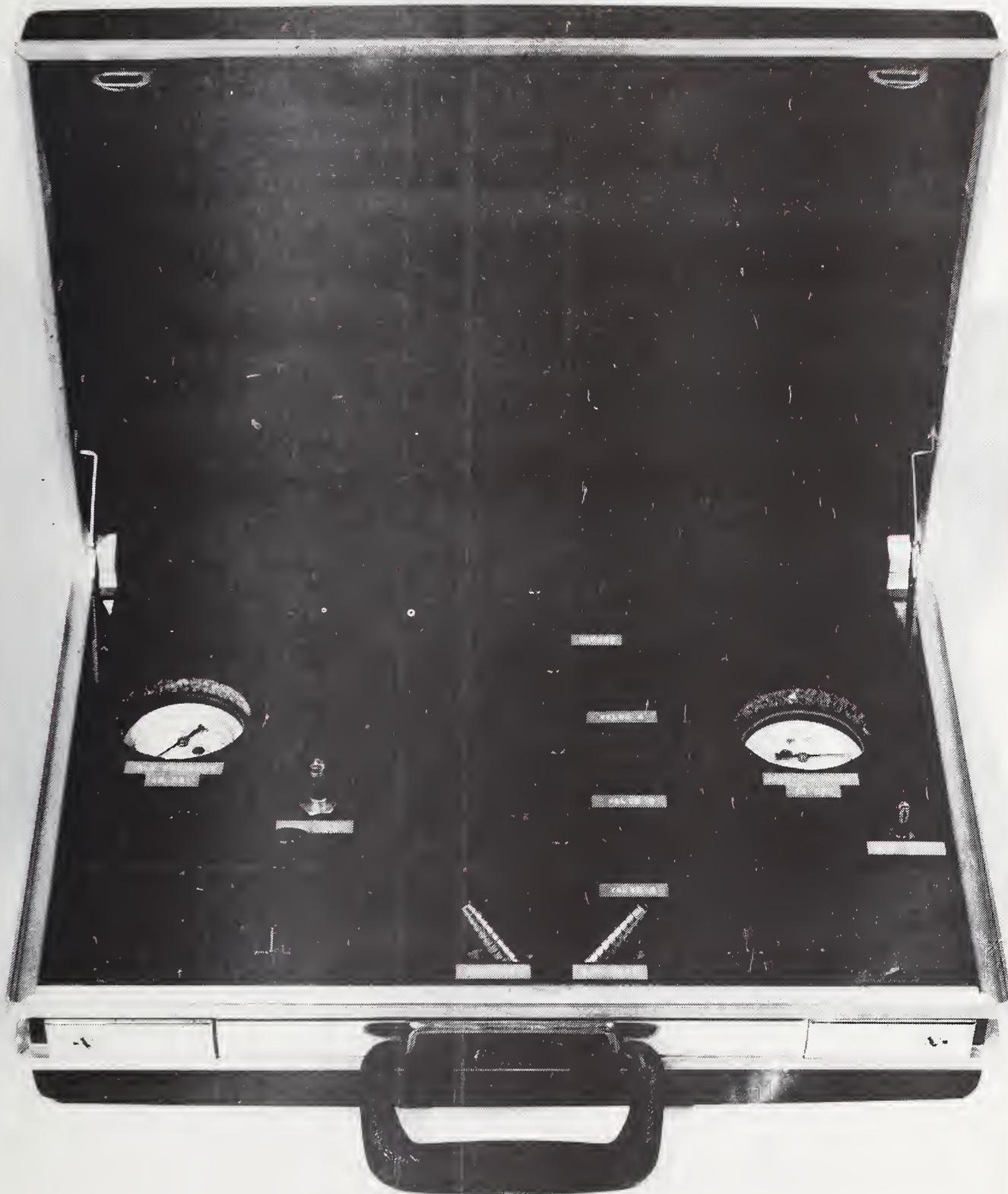


Figure 6



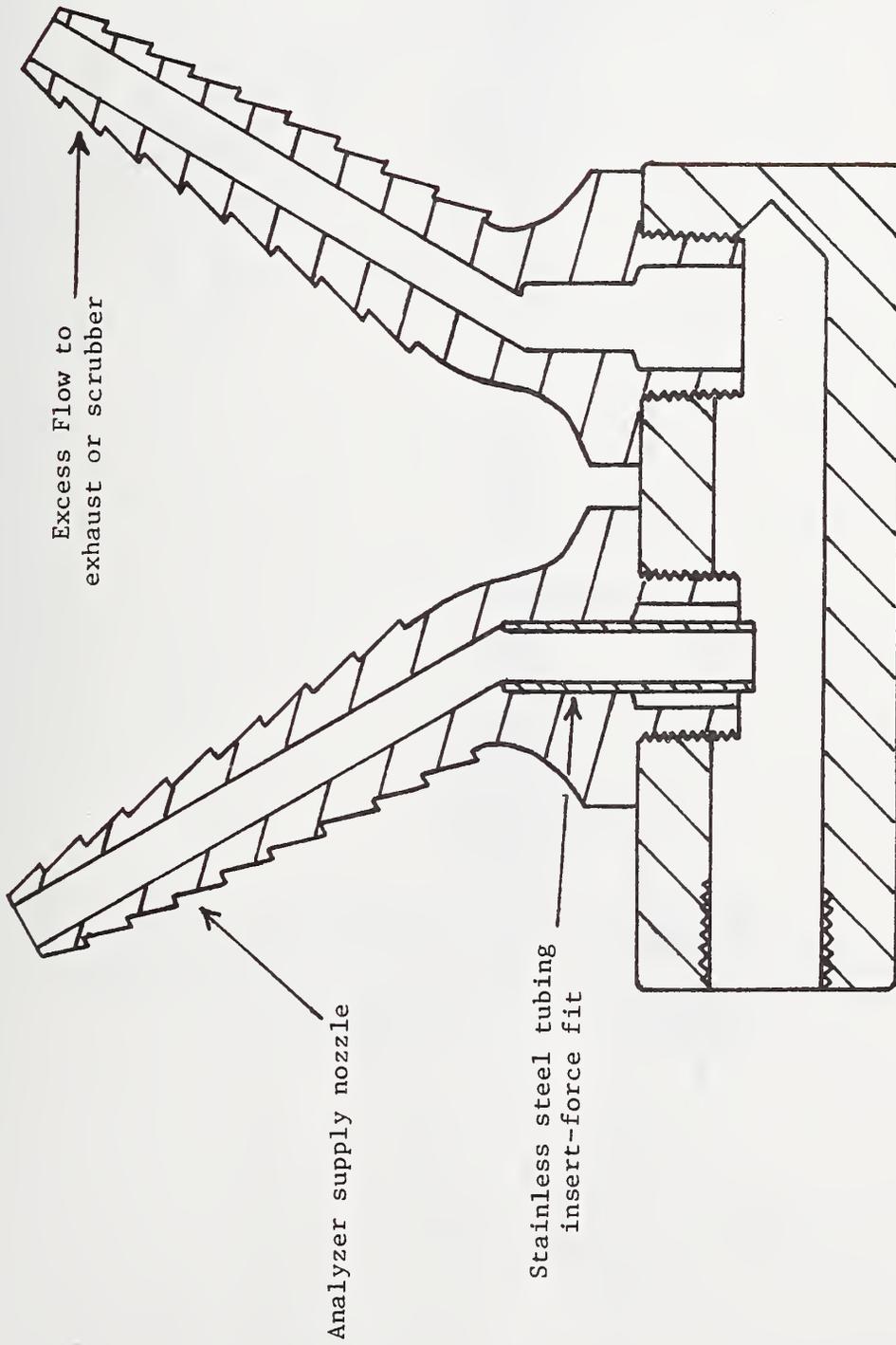


Figure 7 Cross Section of Outlet Manifold

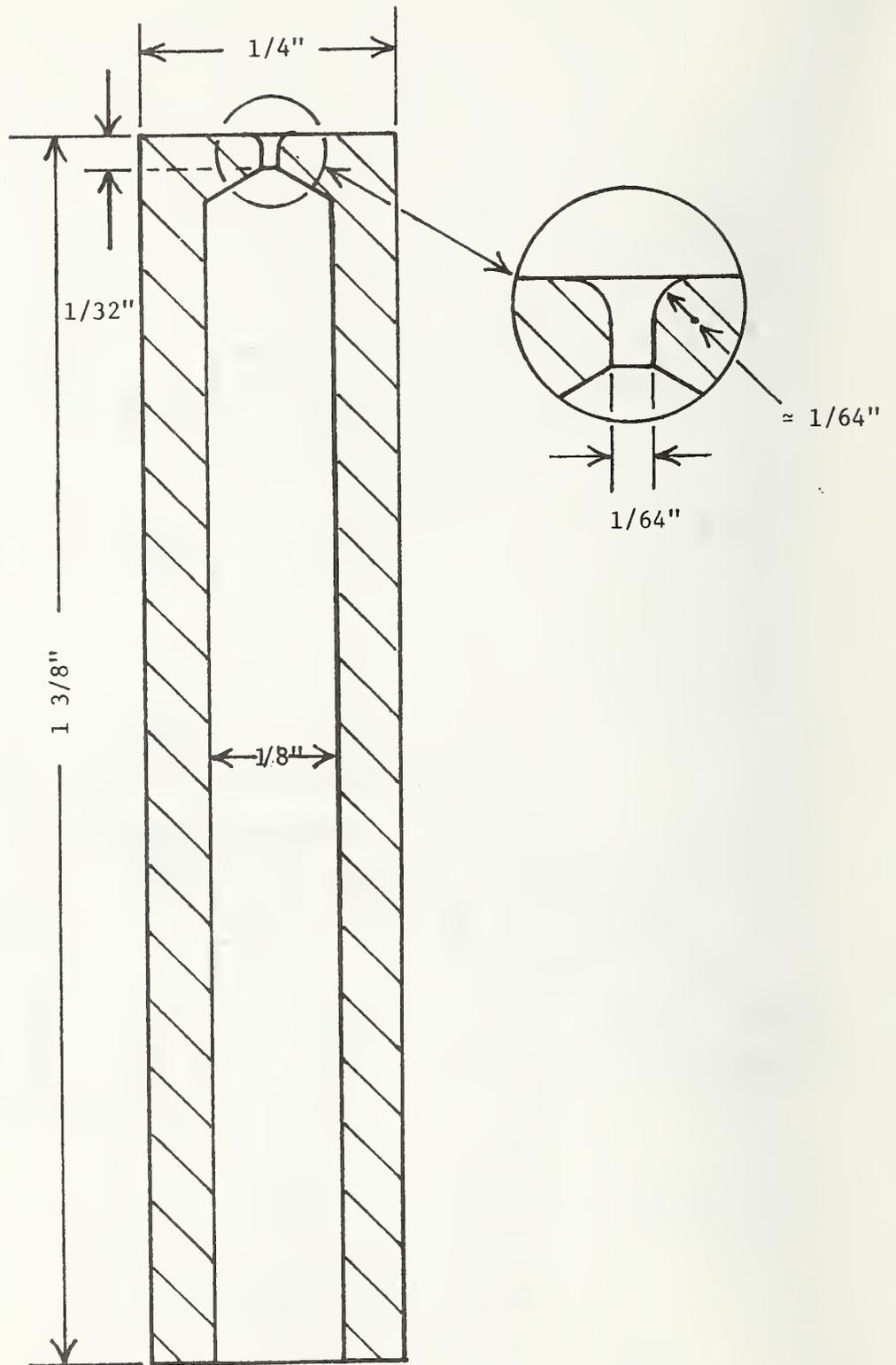


Figure 8 Cross Section of Critical Flow Nozzle

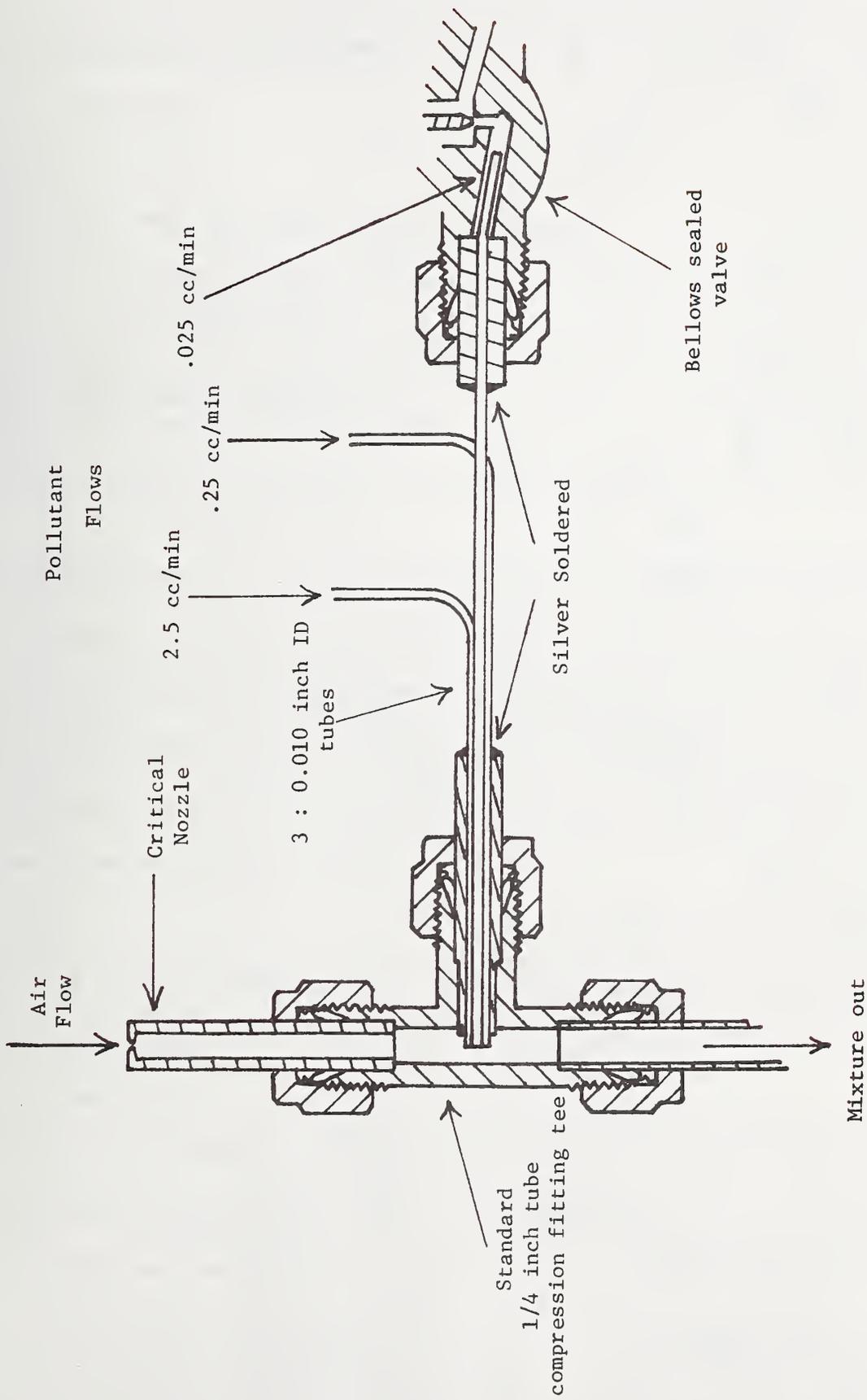


Figure 9 Cross Section of Mixing Region and Capillary Tube End Fittings

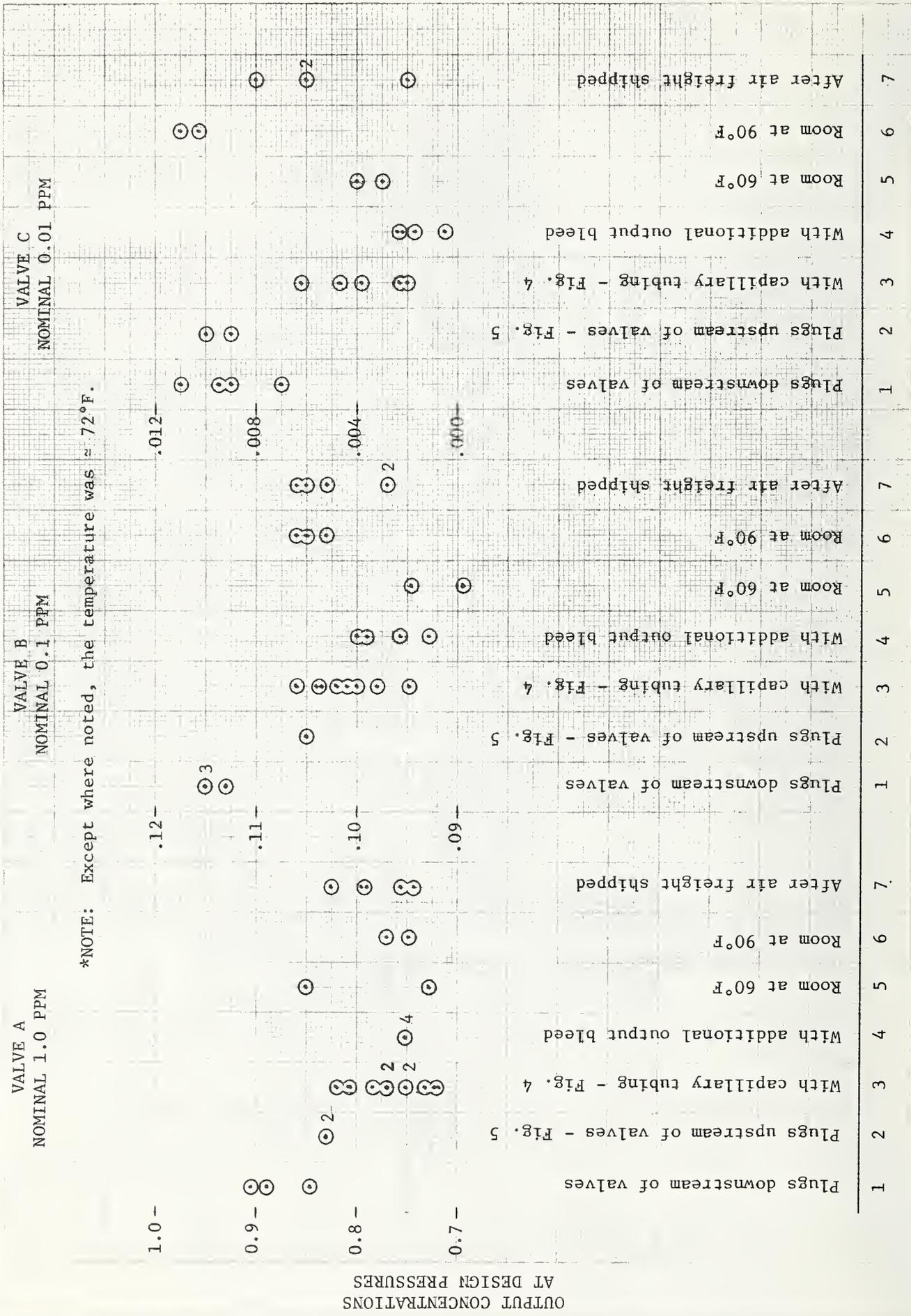


Figure 10 - SEQUENCE OF TESTS

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		14. Sponsoring Agency Code	
15. SUPPLEMENTARY NOTES			
<p>16. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.)</p> <p>The building and evaluation of a prototype SO₂ polluted air delivery system (PADS) is discussed. The delivery system was built to delivery sulfur dioxide (SO₂) in air at a rate of 5 liters per minute with design concentrations by volume of 1.0, 0.1 and 0.01 parts per million. It consists of a diluent air delivery system utilizing a critical flow sonic nozzle and three separate concentrated SO₂ in air flow systems utilizing laminar flow porous plugs, one plug for each desired output concentration. The delivery system is contained in a dispatch case and the two gases are delivered to it from pressurized containers through detachable supply lines. Prospective use by unskilled technicians dictated simplicity and durability and compactness.</p> <p>By maintaining specific upstream pressures on the critical flow nozzle and the laminar flow porous plugs of 45 and 12 psig respectively, the prototype PADS produced average output concentrations of 0.76, 0.100 and 0.003 parts per million of SO₂ in air based on concentration measurements with an NBS calibrated analyzer. The expected output concentrations were 0.98, 0.105 and 0.010 respectively, based on flow calibrations of the individual components. The uncertainty of the output concentration is estimated to be about ± 10 percent.</p>			
<p>17. KEY WORDS (six to twelve entries, alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons)</p> <p>Air pollution; critical flow; laminar flow; nozzle; porous plug; sulfur dioxide concentration</p>			
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