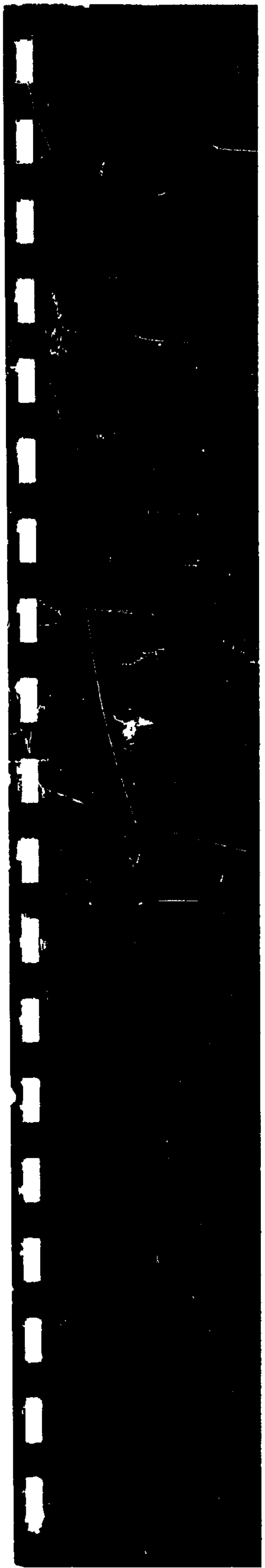

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DA 18-108-CML-7006

FINAL REPORT

Covering the Period
22 December 1961 to 15 May 1963

INHALATION AEROSOL DOSIMETER

Prepared by
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June 1963

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FOREWORD

This report was prepared by the MRD Division of the General American Transportation Corporation (GATC), Niles, Illinois - to summarize the work accomplished under Contract DA 18-108-CML-7006 for the development and construction of an instrument to sample, record and integrate airborne particulates being inhaled and exhaled by experimental subjects. This work was initiated while the MRD Division was part of the American Machine & Foundry and named the Mechanics Research Division. On 1 September 1962 this Division was purchased by GATC and renamed the MRD Division; Contract DA 18-108-CML-7006 was included in the sale.

The work reported herein was initiated on 22 December 1961 and completed on 15 May 1963. Mr. C. L. Punte of the U. S. Army Chemical Research and Development Laboratory served as Project Officer for this contract. The authors sincerely appreciate the technical advice and guidance provided by Mr. Punte, and the assistance of Mr. E. L. Cooper of MRD in designing the electronic portion of the Inhalation Aerosol Dosimeter developed, constructed and then installed at the Army Chemical Center, Maryland.

This report is cataloged by the General American Transportation Corporation as Report MRD 1181-70.

ABSTRACT

An Inhalation Aerosol Dosimeter capable of continuous measurement and monitoring of respiratory retained amounts of aerosols has been developed. The instrument is capable of measuring and monitoring concentrations of aerosols in the range of several hundred to few micrograms per liter of air, and in the particle size range from 10 microns to any submicron size range desired.

Laboratory studies of various aerosol sensing concepts were made. These studies lead to the selection of the hydrogen flame-ionization principle as the optimum method from the standpoint of reliability and sensitivity.

A novel version of a dual hydrogen flame-ionization detector, operating under slightly reduced pressure has been designed and built in this laboratory.

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SECTION 1

INTRODUCTION

1.1 Program Objectives

The objective of this program was to develop an instrument that would continuously measure and record the amounts of aerosols that are retained by man within his respiratory tract in the course of breathing aerosol laden air. The specific requirements set out in the contract were as follows:

(a) The sampling rate at the point of inhalation and exhalation should be 1 to 5 liters per minute, taking into account the subject's respiratory volume (0.5 to 3 liters), respiratory rate (5 to 30 per minute), and respiratory tidal volume (200 to 1000 cc).

(b) Airborne particle size to be sampled are 0.2 to 10 microns in diameter.

(c) Aerosol concentrations to be inhaled are 10 to 200 micrograms-per-liter and exhaled are 1 to 200 micrograms-per-liter.

(d) Type of particles to be sampled are either liquid or solid.

(e) Aerosol impaction and deposition on inlet and outlet valves must be negligible.

(f) The instrument should provide a constant record of retained dose administered (0.001 to 10 mgs).

(g) The instrument response should be within 10 seconds.

The tasks set out by MRD to attain these objectives were as follows:

(1) Determine the most effective technique for measuring, monitoring, and recording aerosol content.

(2) Prove out the selected components and the entire system by testing.

(3) Design and fabricate an instrument that operates on the selected principle, using proven components.

(4) Test the instrument in the manner prescribed by CRDL.

1.2 Background

The prior methods (Reference 1)[*] used for measuring and monitoring the quantity of aerosols retained by man within his respiratory tract were based upon periodic measurement of the concentration of the aerosol in the inhale stream, allowing the subject to breath this aerosol laden air for certain periods of time, and then measuring the total amount of aerosol exhaled by the subject. On this basis it was tentatively possible to establish the dosage of the aerosol retained by the test subject.

The inhale concentration was measured by scrubbing a portion of the inhaled air (measuring the volume) and then analyzing the content of the aerosol colorimetrically. The total amount of aerosol exhaled was determined by placing an electrostatic precipitator on the exhale side and then analyzing the content of aerosol by the same colorimetric method.[**] The volume of air inhaled was measured with the aid of a spirometer.

With this setup it was a rather lengthy procedure to administer a predetermined dose of the aerosol to the test subject, and this problem lead CRDL to seek the development of an advanced version of an Inhalation Aerosol Dosimeter (IAD).

The concept originally proposed by MRD was based upon the utilization of thermal conductivity principles as the aerosol sensing method.

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[*] See page 63

[**] Bromsulphalein (BSP) aerosol was used throughout in the tests and its concentration was measured at 580μ in alkaline solution.

The overall Inhalation Aerosol Dosimeter, as originally proposed by MRD is shown in block diagram in Figure 1. At that time it was also planned to use a sampling valve to take samples of the "inhale" and "exhale" air and sweep it into the analyzer by a carrier gas. A conceptual design of the sampling valve is shown in Figure 2.

The aerosol was to be generated externally and supplied into the IAD. The sampling valve is actuated by a sensitive pressure switch and identical samples (on the basis of volume) are injected simultaneously after preheating into the detector cell. Two signals are picked up by the attenuator, one of the signals is inverted and opposed to the other. The resulting signal is fed into the recorder; it is integrated and read out digitally. At the same time the cumulative amount of aerosol retained is established by coordinating this signal with that representing the actual volume of air inhaled. While in the course of developing the instrument some changes were made, the original concept of the overall dosimetry system as shown in Figure 1 remained the same.

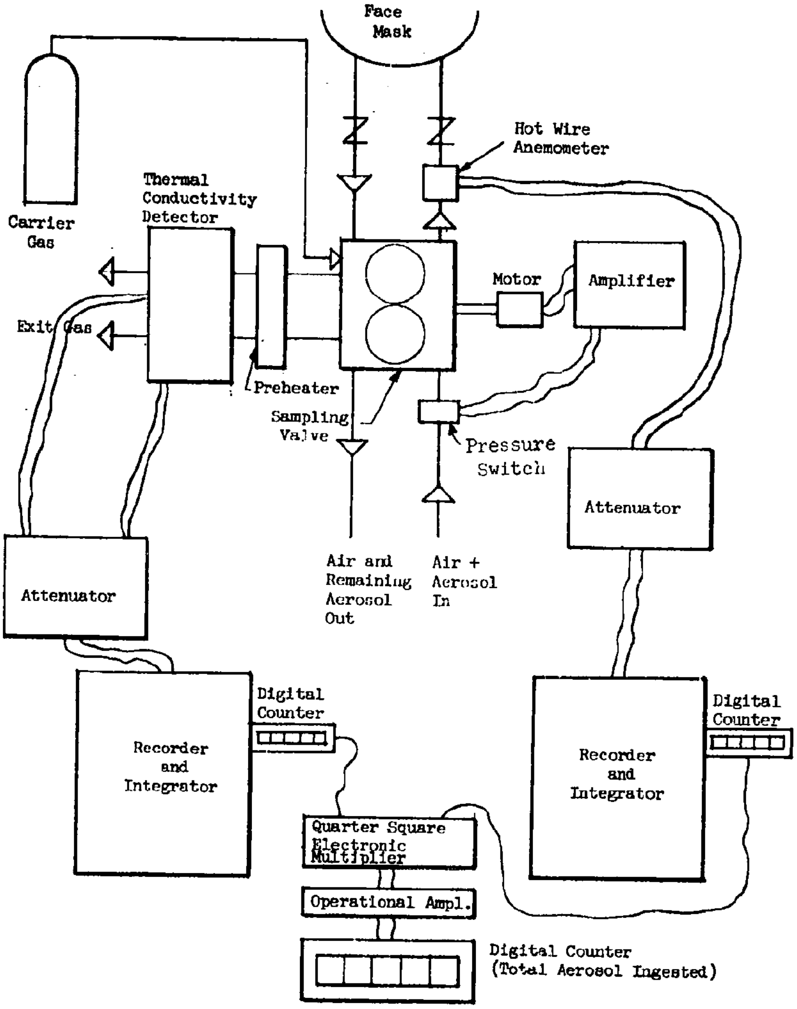

Figure 1 BLOCK DIAGRAM OF THE INHALATION AEROSOL DOSIMETER AS ORIGINALLY PROPOSED BY MRD

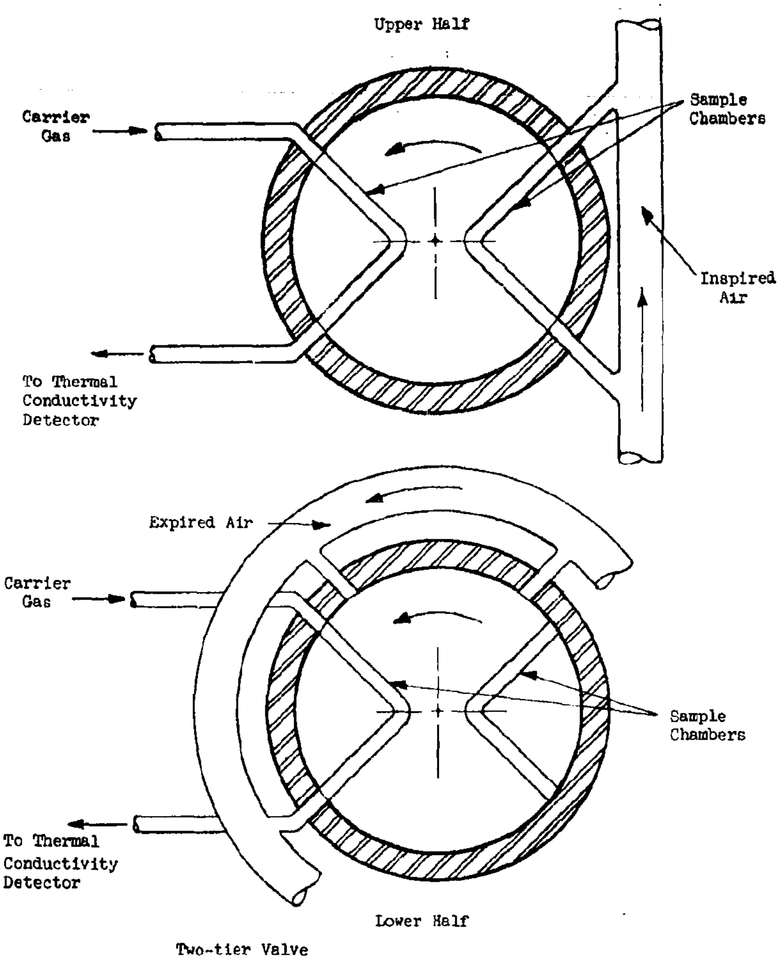

Figure 2 CONCEPTUAL DESIGN OF SAMPLING VALVE

SECTION 2

ENGINEERING SURVEY

The purpose of this survey was to determine the best and most practical detection techniques. Thermal conductivity was the most likely detection technique as indicated in the Introduction and thus is discussed first; subsequently hydrogen flame-ionization and other methods are discussed.

2.1 Thermal Conductivity

The thermal conductivity cell, better known by the name of katharometer was first introduced by Shakespear (Reference 2). It was designed primarily for the measurement of the concentration of various gases on the basis of their difference in thermal conductivity. Its widest application is made in gas chromatography. With time many improvements were made, and currently commercially available katharometers are capable of measuring samples in the range of 10^{-7} to 10^{-8} grams.

2.1.1 Theory

The operation of a katharometer is based upon comparing the thermal conductivity of an unknown gas with that of a standard or reference gas by measuring change in electrical resistance as a function of temperature as shown in Figure 3. As the thermal conductivity of the reference gas is known, it is a simple matter to calculate the absolute conductivity of the unknown gas from (a) the measured relative conductivity and (b) the known absolute conductivity of the reference gas. Figure 3 shows two similar current-heated filaments in two similar cells, and two similar inert resistances. These two elements are connected to a Wheatstone bridge. If the reference cell contains

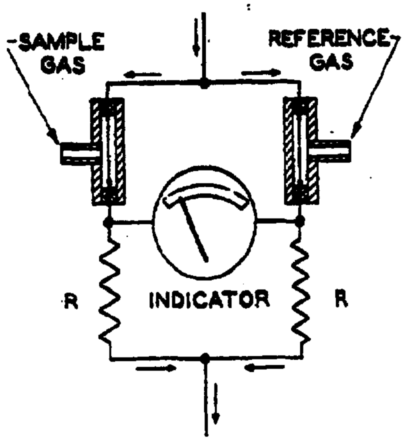

Figure 3 PRINCIPLE OF THE OPERATION OF A KATHAROMETER

air and the sample cell contains a gas having a different thermal conductivity, the two hot wires will not be at the same temperature; and they will not have the same resistance. The temperature difference will be inversely proportional to the thermal conductivity of the gas for a given power input to the hot wire. A good rule is that the thermal conductivity of the gas varies inversely with its density; for example, the lightest gas, hydrogen, has the highest thermal conductivity. The bridge then will become unbalanced to an extent depending on the difference in conductivity. The extent to which the bridge becomes unbalanced will depend also on the percent composition of the sample, and the scale on the indicator can be calibrated in terms of percent composition.

The Wheatstone bridge is universally used in katharometers. The use of four active filaments, such as shown in Figure 4, increases the sensitivity to a given change in composition. These filaments are matched for temperature coefficient of resistance, and are usually of tungsten or platinum.

It is not always necessary to know the absolute thermal conductivity of the unknown. Often it is desired to know the difference in thermal conductivity produced by a given change in the composition of the mixture. This difference can be established empirically. It is this change in aerosol concentration between the "inhale" and "exhale" side of the breath that is basic in the Inhalation Aerosol Dosimeter. This change represents the amount of aerosol retained by the test subject.

2.1.2 Application

The quantity of heat conducted by a gas varies with the nature of the gas, extending from poorly conducting (having one-fifth the conductivity of air) to hydrogen (having seven times the conductivity of air). Gas and vapors of any conductivity between these two extremes can be analyzed accurately by proper selection of the reference gas.

The simplicity of operation of the katharometer, and expecially the commercial availability of cells capable of operating at 450°C lead to the selection of this method. As shown in Figure 2, a predetermined volume of both the inhaled and exhaled air was to be collected and held within the specially designed sampling valve and then simultaneously injected into the katharometer. Since the same carrier gas was to be used and all the other variables were held constant, the signal obtained from the cell would be directly proportional to the difference in concentration of aerosol in the two samples.

It was further believed that non-vaporizable aerosols in the micron and

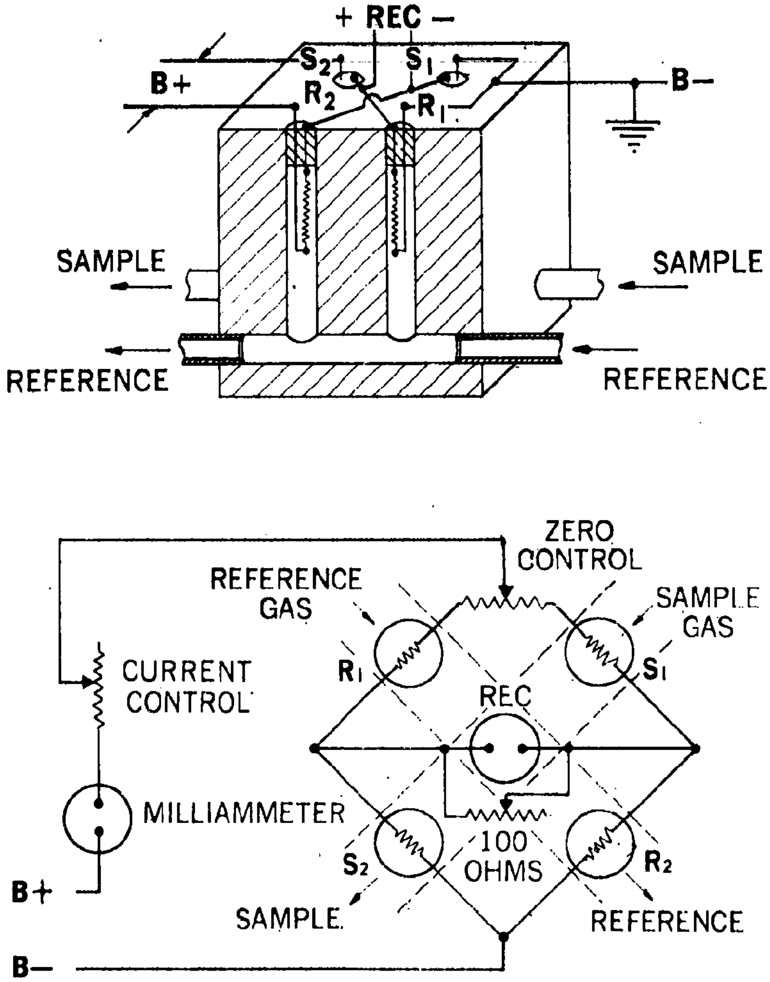

Figure 4 FOUR FILAMENT CELL AND ITS BASIC CIRCUIT

submicron particle size range would affect the hot filaments in a similar manner as the gaseous substance.

A katharometer capable of operating at 450°C was purchased from Gow-Mac Instrument Co., Madison, N. J. to verify the above assertions. The experimental results obtained are discussed in Section 3.

2.2 Hydrogen Flame-Ionization (HFI)

2.2.1 Theory

Gases burning in a flame become sufficiently hot for a small proportion of the molecules to acquire sufficient energy to ionize and to give the flame an electrical conductivity. When an organic vapor enters the flame, the conductivity becomes greater and the increase in conductivity may be measured and recorded. This is the basis of the hydrogen flame-ionization detector.

At least two reasons have been suggested for the increase in conductivity. One is that the ionization is purely a result of the high temperature in the flame, and that organic compounds, having smaller ionization potentials than hydrogen, produce more ions at a given temperature than does hydrogen (Reference 3). Another is that particles of carbon formed in the flame have a small work function, and thus supply a large number of electrons to the flame (Reference 4). However, neither of these explanations are adequate to account for the observed conductivity, as the ionization efficiency of the hydrogen flame is small, and it has been quoted as 0.01 to 0.05% (Reference 5) and in many cases even smaller than this.

Figure 5 shows the basic circuit of the flame ionization detector. HFI has found widest application in gas chromatography where the effluent from the chromatographic column is passed into the flame whose electrical conductivity is being measured. The simplest arrangement is to use the metal jet as the cathode and the screen (or rod, or ring) above the flame as the anode.

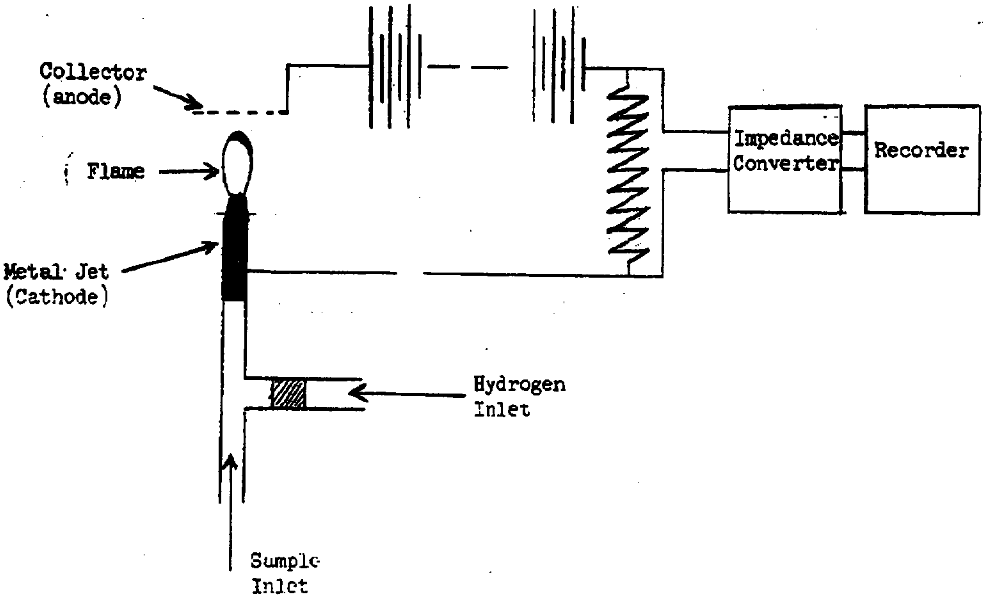

Figure 5 BASIC CIRCUIT OF HFI DETECTOR

The voltage applied across the flame is usually 200 to 300 volts DC. This provides saturation voltage, that is, all available ions arrive at the electrodes, so that further increases in voltage do not increase the current. The design of the detector should be such that the negative electrode (the flame jet) should not become too hot, as it may emit thermal electrons which raise the background conductivity, and raise the noise level. This may happen if a small metal jet of low heat capacity is used, or if the collector is extended too far into the flame. Another important factor is that the electrodes should not be too far away from the flame, or the ions will recombine before reaching it, so that the current will be too small.

The resistance of the hydrogen flame is generally of the order of 10^{14} ohms, and the current through the cell varies from the order of 10^{-12} amp background current to about 10^{-7} amp on maximum signal. Different authors using different detector designs give remarkably similar figures for the limit of detection, i.e., the concentration equivalent to noise. This limit appears to be about 1 in 10^{9} moles for substances in hydrogen (References 6, 7, 8).

The background current produced by the pure hydrogen flame is proportional to flow rate of hydrogen, and thus <u>variation</u> in the flow rate appears as background noise and, in practice, it appears that this factor determines the noise (Reference 7).

Ongkiehog (Reference 7) gives the following relation between the vapor concentration equivalent to the noise produced by uneven flow:

$$\Delta = d \cdot \frac{S_h}{S} \cdot \frac{F_h}{F_g}$$

where Δ = vapor concentration equivalent to noise

S = response of vapor (amp gram^{-1} second)

S_h = response of pure hydrogen (amp gram^{-1} second) calculated from background current of pure hydrogen flame

F_h = hydrogen flow rate

F_g = carrier gas flow rate; F_h if hydrogen is the carrier gas and no mixture is made

d = flow fluctuation in proportion to total flow

The linearity of the detector is claimed to be from about 10^{-9} moles (limit of detection) to a maximum concentration of about 5×10^{-3} moles (Reference 9), or a dynamic range of about 10^{6}. A high impedance amplifier is used to amplify the resulting signal.

2.2.2 <u>Application</u>

The application of HFI for the detection and measurement of aerosols appeared to be straightforward. Either the aerosol could be vaporized, as discussed in

Section 2.2.1, or the aerosol particles could be brought directly into the flame and allowed to ionize. It has been reported (Reference 6, 10) that the flame is extremely sensitive to fine dust particles so that both combustion air and the hydrogen must be filtered before it enters the detector. Apparently it is this phenomenon that makes it possible to measure both organic and inorganic particulates in the air.

In general, there is a tendency for molar response (e.g., measured in coulombs per mole) to increase linearly with increasing carbon number. Such behavior is generally true for straight-chain hydrocarbons, e.g., the signal obtained from the same volumes of hexane is twice the signal obtained from propane (Reference 7).

Desty, et al (Reference 8) found that the response obtained using 2:2:4 - trimethylpentane is about 30% greater than for ethylbenzene, and 15% greater than for n-octane, all C_8 molecules.

The available literature deals essentially with gases and vapors and very little information is available on the detection and measurement of the concentration of aerosols. At this point it may be only theorized (and this is to be proved) that it may be possible that the detector signal response is directly proportional to the number of particulates fed into the flame, especially if the particulates are not previously vaporized.

2.3 Other Methods

Numerous other methods were also investigated, including surface potential, β-Ray ionization, D.C. and R.F. discharge, and photometry.

2.3.1 Surface Potential Detector

The surface potential detector operates on the principle of generating an AC current when the gas is passed between two metal plates, one of which is covered with a monomolecular layer of a substance which will absorb the vapor (or aerosol) in question. Since one of the plates is vibrated mechanically there is a constant alternating EMF which changes when the substance in question is adsorbed on the surface of the plate. The sensitivity is claimed to be in the range of 1 part ethyl oxalate in 10^6 parts nitrogen (Reference 9).

This method was disregarded for the application of aerosol detection because the adsorption and desorption rates on the plate are too slow; for example, the complete detector analysis cycle for ethyl oxalate is about 5 minutes.

2.3.2 β-Ray Ionization

This detector is based on the principle of measuring the current passing through a gas ionized by β-ray radiation (Reference 11). For purposes of explanation, one may regard the β-rays as streams of particles emanating from the source. These particles traverse the gas, and after traversing a distance the average length of which is the mean free path, they collide with an atom or molecule (or the aerosol particle) of the gas. These rays are emitted from the radioactive material with a large energy which is sufficient to displace an electron upon collision.

The energies with which ionizing particles are emitted from radioactive materials are of the order of 10^4 to 10^6 ev, whereas the energy required to ionize singly an atom or molecule is of the order of 10 ev. Thus, after one collision, the ionizing particle has lost very little energy, and it can then go on to collide with another atom, produce another ion and electron, and so on until it is finally brought to equilibrium.

The range of β-radiation from Sr^{90} (0.54 Mev) in air is approximately 2.7 meters (Reference 11) so that the possible health hazard must be considered in designing the detector. It was for this reason that this principle was disregarded since it was desirable that the mask assembly be situated close to the detector.

2.3.3 D.C and R.F. Discharge

This detector is based upon the principle that when an R.F. discharge occurs between two electrodes wherein the field is diverging, a D.C. potential results. This potential is sensitive to the composition of the gas through which the discharge is passing (Reference 13). This detector has not been used widely, and apparently is not available commercially so that its operation with aerosols could not be evaluated.

2.3.4 Photometry

Photometry is perhaps the most widely used in investigating aerosols. This technique is based upon comparing the light intensities obtained from passing the light through an aerosol sample and an aerosol free sample. This difference in intensity is proportional to the difference in concentration of the aerosol in the two samples. This method has been used successfully with aerosols of one micron or larger diameter size, but it is limited in its application when smaller aerosol particles are considered, and essentially is not applicable for particles of 0.3 microns or less in diameter (Reference 14). For this reason this technique was not evaluated in any greater detail.

2.4 Conclusion

The available literature describes various types of detecting devices for the detection of gases and vapors, but very little information is available on the detection of aerosols. Of the various candidate detection techniques, thermal conductivity or katharometry and hydrogen flame ionization appeared to be the most promising. While thermal conductivity is limited in its sensitivity, it is a well established and proven technique. Numerous detectors operating on this principle are commercially available.

Hydrogen Flame-Ionization technique, on the other hand, is one of the most sensitive and simplest techniques yet developed for the detection of organic constituents in air. This technique has found extremely wide application in gas chromotography, and its application is constantly growing. Because of its high sensitivity and the fact that numerous components and detectors utilizing this principle are commercially available, it was decided to analyze this method in detail in addition to that of thermal conductivity.

SECTION 3

EXPERIMENTAL EVALUATION

This section presents the experimental evaluation of the concepts of thermal conductivity and hydrogen flame-ionization, which an engineering survey indicated as the most applicable for detection and measurement of the concentration of aerosol in the air. While the two approaches were studied and evaluated during the same time period, thermal conductivity will be discussed first, as this was the method originally proposed.

The aim of this experimental evaluation was to establish the best detection method which would then be used in the Inhalation Aerosol Dosimeter.

3.1 Thermal Conductivity Cell (TC)

In the initial feasibility studies for measuring the concentration of aerosol in air, a Beckman Instrument Company GC-[illegible] Gas Chromatograph equipped with their standard TC cell was used. In this evaluation, the chromatographic column was removed, and a direct link using 1/8 inch stainless steel tubing was made between the TC cell and the sample inlet port. The cell was operated at 240°C, the upper temperature limit.

Initial results obtained using bromsulfalein aerosol in the concentration range of about 100 micrograms per liter indicated only a slight response of the cell - about 10% of total indicator scale at optimal sensitivity settings. At this point it was thought that the TC cell response could be significantly improved by increasing the operating temperature of the cell and at the same time vaporizing the aerosol prior to its injection into the TC cell.

Subsequently, a new TC cell was purchased from GowMac Instrument Company, Madison, N. J. This is the type of cell as shown in Figure 4, Section 2 of this report. In operating this cell, it was observed that about a threefold increase of sensitivity was obtained as compared with the Beckman cell. At the same time it was observed that this sensitivity decreased rather rapidly as the test progressed. Closer examination of the cell filaments revealed that a thin film was forming over the filaments and this apparently continuously decreased the filaments' sensitivity. Such behavior was most marked when high boiling point aerosol simulants such as glycerine and dibutyl phthalate were used.

As a next step in this investigation, it was attempted to test out the "Glow-plug" principle. This principle is similar to that of TC except the filament is much thicker in diameter and it is capable of operating at much higher temperatures (up to 1100°F). It was thought that at these temperatures the formation of a thin film could be prevented. The test results were unsatisfactory as the sensitivity was considerably below that required in the contract.

3.2 Hydrogen Flame Ionization (HFI) Technique

The HFI detector used in the preliminary evaluation to establish the feasibility of this method was a standard model (Model 60-1) as supplied by Research Specialties Co. of Richmond, California. As pointed out previously, this detector is built to operate under positive displacement, and thus in this application it was necessary to make some modifications. Silicone rubber was used to seal the detector hermetically. While this method of sealing withstood the elevated temperatures for several days, and the results obtained during this time were very good, the silicone rubber began to decompose and the detector background noise increased very appreciably after about three days of continuous operation.

3.2.1 HFI Detector - Laboratory Model

After these preliminary findings, which indicated the feasibility of the HFI technique for detection and measurement of the concentration of organic aerosols, it was decided to construct an HFI detector in this laboratory that would be more applicable for this study.

A schematic diagram of the initial version of the HFI detector as constructed in this laboratory is shown in Figure 6. The crucial problem was to maintain a slightly reduced but very precise pressure within the detector chamber, so that air could be continuously and uniformly sampled and analyzed.

The aerosol is brought in through the capillary tubing and it is mixed with hydrogen prior to entry into the flame. In the flame, ionization takes place (as discussed in Section [illegible]) and an increase in the electrical conductivity of the flame results. The signal is picked up by a collector screen, amplified by the electrometer and fed into the recorder where a permanent record is made.

The initial version of the flow control system is shown in Figure 7. The aerosol sample is drawn into the detector from a main aerosol supply line, where the pressure is approximately atmospheric. The diameter of the main supply tube is about 1 inch while that of the detector sampling tube is about 1/64 inch (capillary tubing).

The effluent gas coming from the detector chamber passes through a series of three flasks, as shown in Figure 7. The purpose of the first flask is to reduce any immediate fluctuation that may result within the detector chamber. This fluctuation may be due to uneven combustion of the gases or actual temperature fluctuation within the detector chamber. The ability to maintain precise and constant pressure within this flask is of utmost importance as it is this reduced pressure that controls

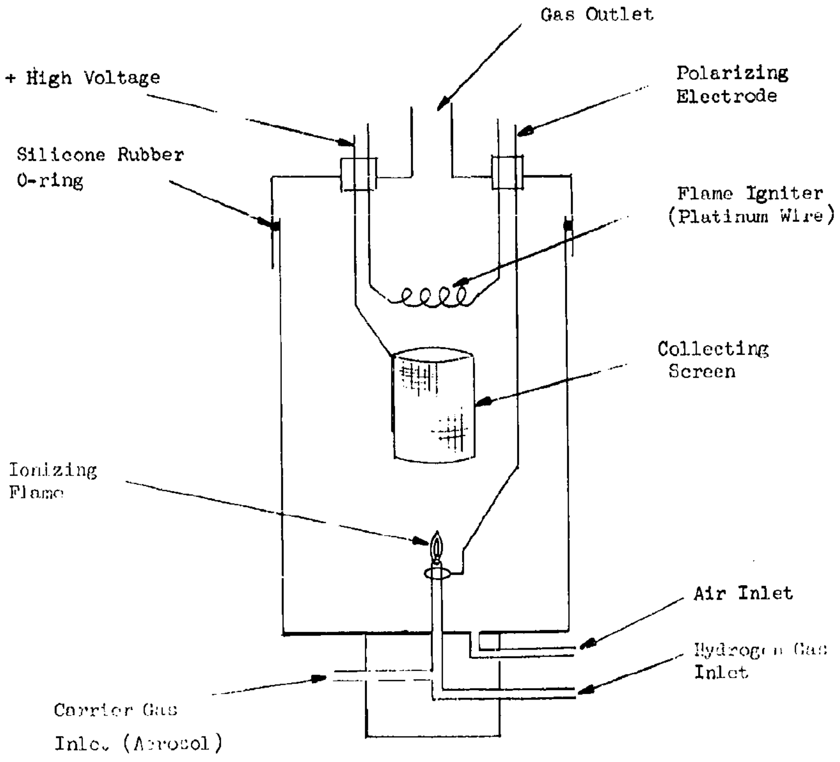

Figure 6 SCHEMATIC DIAGRAM OF THE HFI DETECTOR BUILT IN THIS LABORATORY

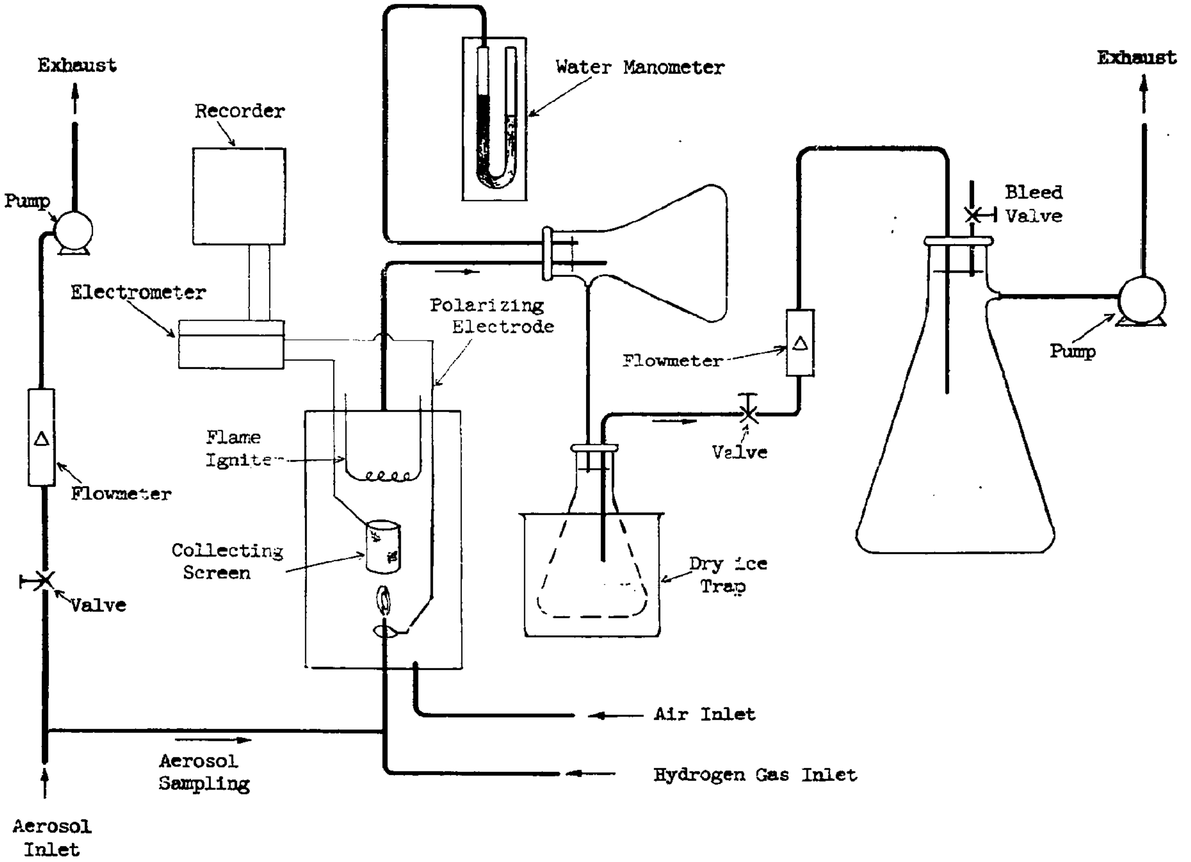

Figure 7 SCHEMATIC DIAGRAM OF THE HFI APPARATUS USED TO ESTABLISH THE FEASIBILITY OF HFI TECHNIQUE

the sampling rate of the aerosolized air into the detector. A water manometer is connected directly to this flask so that variation in pressure could be observed.

The second flask is to remove and entrap water vapor which is a combustion product. A dry ice trap was used in initial evaluations, however, later it was found that salt water with ice mixture was satisfactory. The purpose of this trap is to prevent moisture clogging the flowmeter and tubing which will disrupt continuous flow of the gases and affect the sampling rate of the aerosol.

The third flask essentially provided a "high vacuum" reservoir, and its function was to reduce the fluctuations of the vacuum pump.

With the overall setup shown in Figure 7, it was demonstrated that BSP aerosol could be detected and monitored continuously in the concentration range of about 80 micrograms per liter. However, higher amplification of the signal resulted in significantly increased background noise, which was directly related to the unstable sampling rate. Initially this instability was attributed to temperature fluctuation within the detector chamber and subsequently an experiment was conducted with the detector being partially submerged in a silicone oil bath, as shown in Figure 8. Despite this modification the signal fluctuation and background noise persisted. This study, however, demonstrated that the temperature effect by itself was rather insignificant and the primary cause was the instability of the detector chamber pressure.

3.2.2 Construction of the HFI Detector - Advanced Model

While the study using the laboratory detector was in progress, plans were made to build two detectors of more advanced design. Figure 9 shows an assembly drawing of this detector. Two such detectors were built and bench tested.

In the construction of these detectors it was intended to control the temperature of the block assembly so that if it were necessary to raise the temperature to

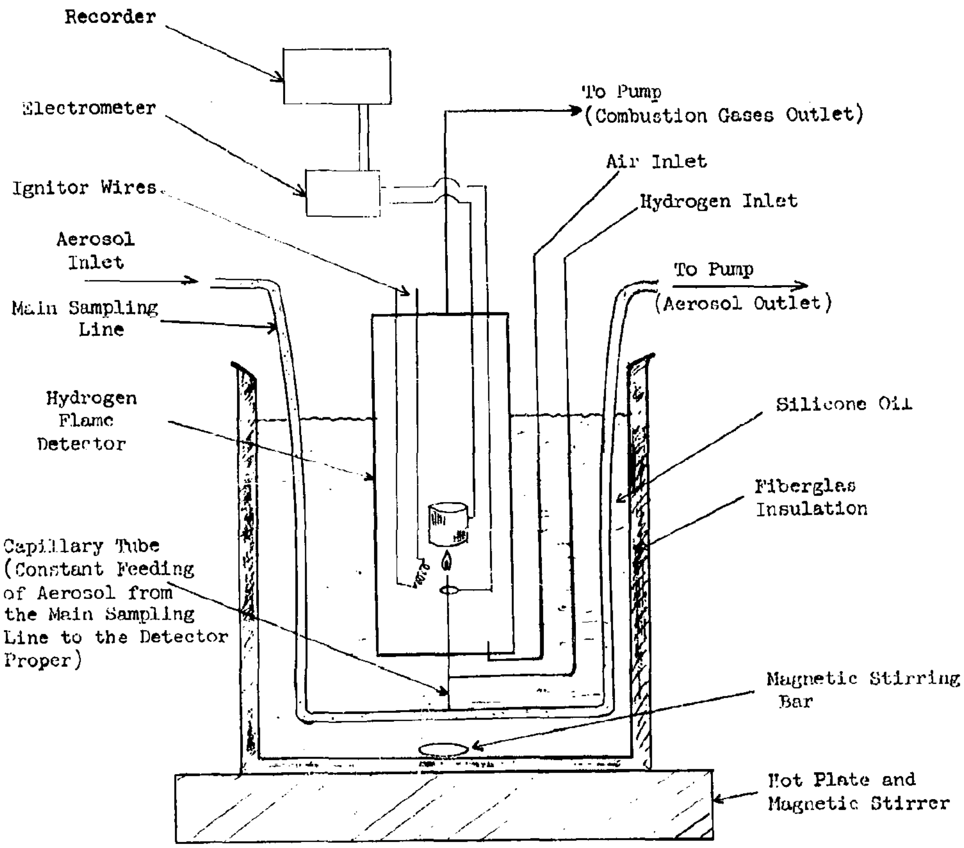

Figure 8 SCHEMATIC DIAGRAM OF HFI DETECTOR PARTIALLY SUBMERGED IN SILICONE OIL

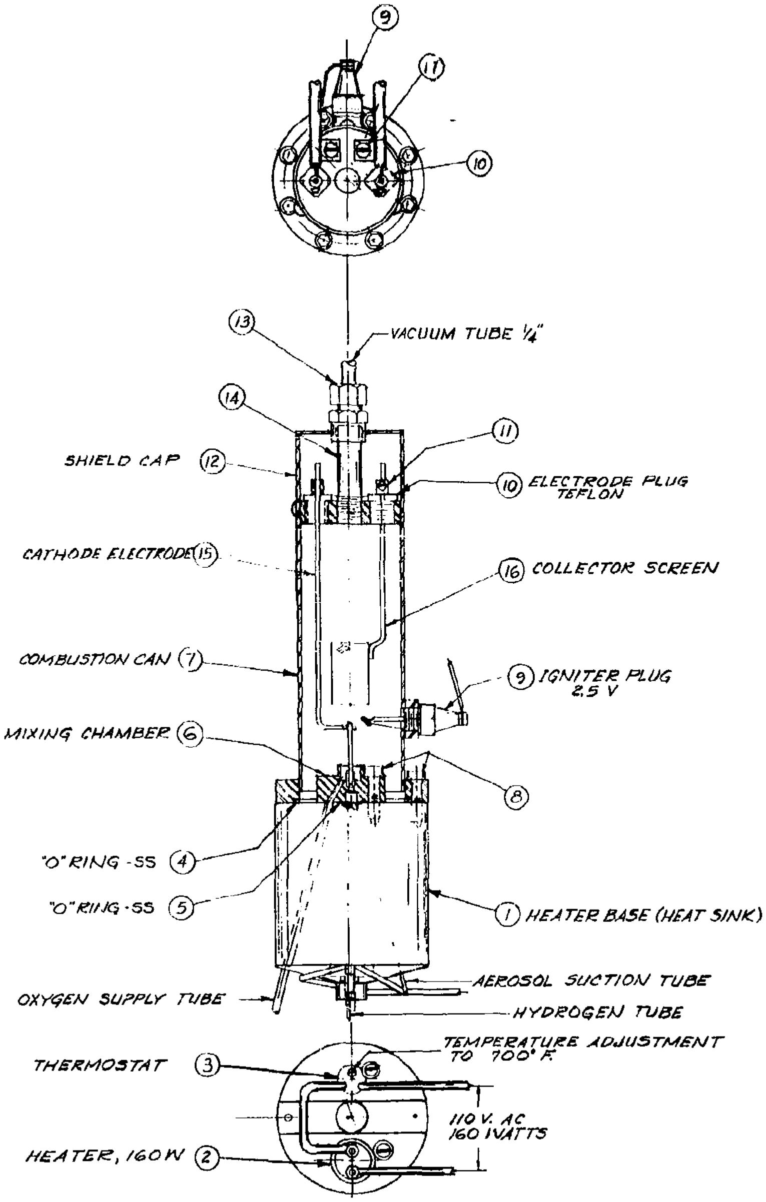

Figure 9 CONCEPTUAL CONFIGURATION OF THE MODIFIED HFI DETECTOR

vaporize the aerosol it could be easily preset. This was done by imbedding a cartridge type-thermostatically controlled heater into the block base assembly as shown in Figure 9.

Figures 10 and 11 show photographs of the bench testing assembly. These two detectors were connected to two separate pumps. In the course of testing, it became evident that with separate flow control systems and using the setup as shown in Figure 7, it would be impossible to establish identical aerosol sampling rates for both detectors.

Another problem that appeared as the testing progressed was that the stainless steel "O" rings, sealing the detector chamber with the base, collapsed and in-leakage of room air resulted. The background noise increased very significantly. While these detectors were [illegible] in actual operation, they provided enough information for the subsequent design and construction of a dual type [illegible] detector. At this point two basic problems had to be solved before any additional steps in this study could be taken.

The first problem was to modify or redesign the two available HFI detectors, and the second problem was to establish a reliable method of maintaining precisely the same pressure within the two detectors, so that the aerosol sampling rate could be virtually the same for both detectors.

3.2.3 Construction of the Final Detectors

An overall appraisal of the existing detectors indicated that it would be more advantageous to red-sign and construct a new detector in such a way that the exhaust of both detectors would be linked to a single vacuum control assembly. Figures 12 and 13 show schematically the construction of this dual detector. Figure 14 shows the shop drawing of this dual detector. Note that the two detectors are enclosed in a single container where the temperature is maintained

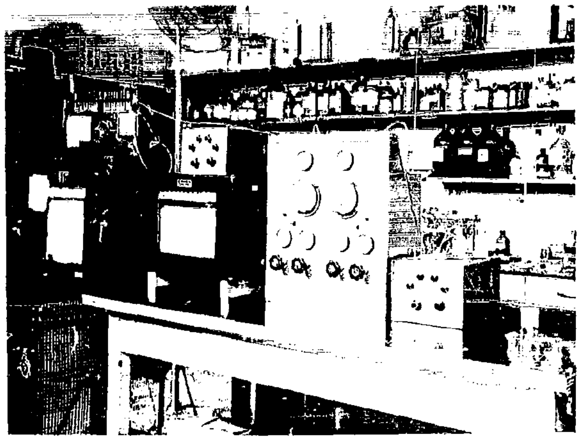

Figure 10 FRONT VIEW OF THE BENCH ASSEMBLY OF THE INHALATION AEROSOL DOSIMETER

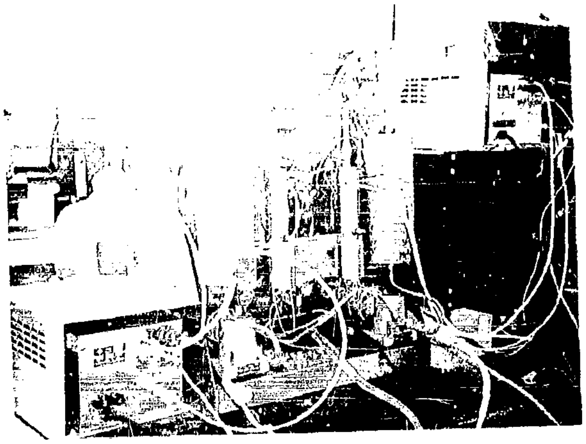

Figure 11 REAR VIEW OF THE ASSEMBLY AS SHOWN IN FIGURE 10

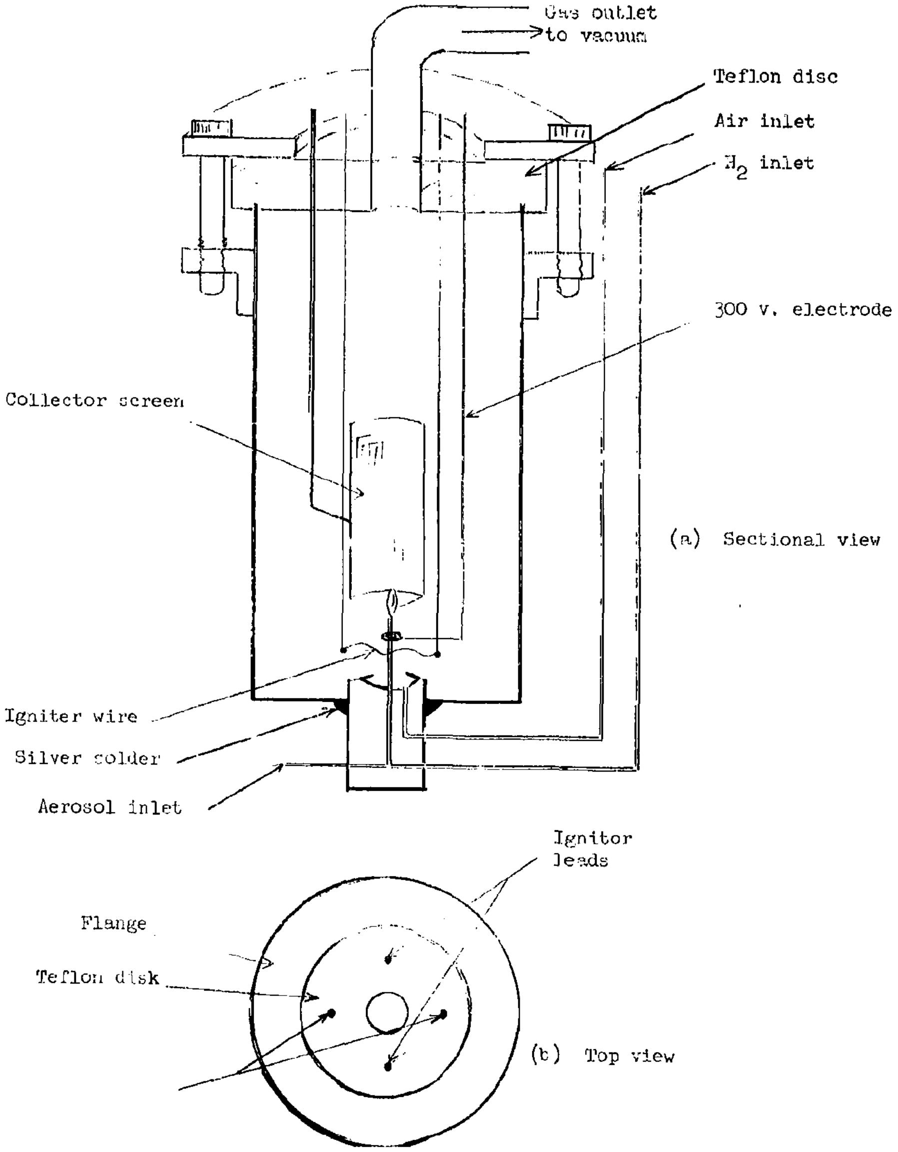

Figure 12 SCHEMATIC DIAGRAM OF THE FINAL MODEL OF HFI DETECTOR (SINGLE CHAMBER)

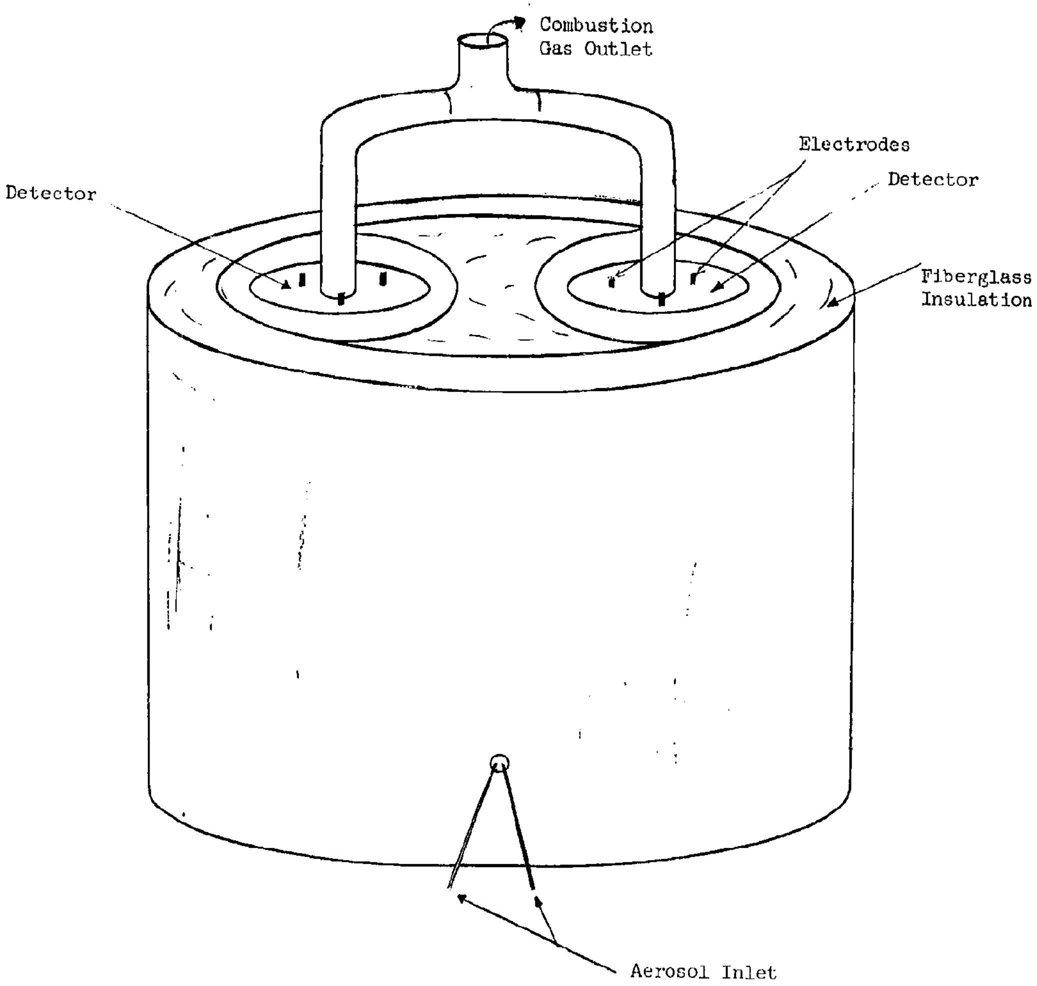

Figure 13 ASSEMBLED TWO HFI DETECTORS, ENCLOSED IN A SINGLE BOX WHOSE TEMPERATURE IS CONTROLLED WITH THE AID OF TAPE HEATER AND VARIABLE TRANSFORMER

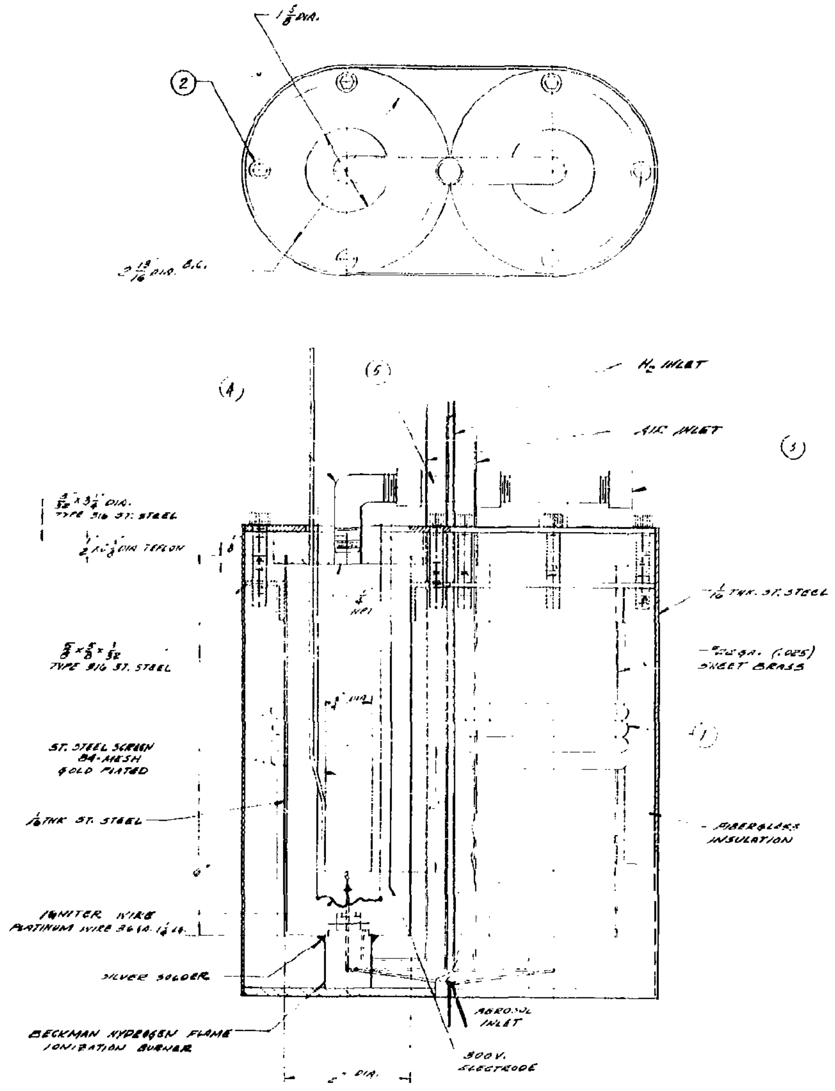

Figure 14 SHOP DRAWING OF DUAL HCl DETECTOR

constant with the aid of a Briskheat Tape heater and a variable transformer.

The detector is separable into two main parts: the base, and the electrode assembly. These are fastened together by means of a flange and four screws.

The base of the individual detector contains the three gas inlet tubes and a ceramic insulated burner tip. The two tubes protruding upward are for hydrogen and the O_2-N_2 mixture, respectively. The third capillary tube is for intake of the aerosol sample. The electrodes are imbedded in the Teflon disk which is sealed to the stainless steel cylinder (part of the base assembly) by means of the flange and the four screws mentioned above.

Negative high voltage (300 volts) is connected to the burner tip (cathode) by means of a small circular washer silver soldered to the cathode. The platinum igniter coil is situated close to the burner tip; one end of it is grounded, the other is actuated by depressing the FLAME IGNITER button on the electrometer panel. The anode or the collector screen operates at or near to ground potential.

Electrical connections are made to the electrode (including the igniter) at the top of the detector. The "high voltage" (300 volts) cable also supplies separately the 2.5 volts A.C. for the igniter coil. Both cables are electrically shielded and Teflon insulated. The complete detector assembly is electrically shielded by placing it into a rectangular aluminum box.

3.2.4 Flow Control

The problem of flow control was subsequently solved through the use of much larger ballast tanks and the use of a very sensitive subatmospheric pressure regulator as supplied by Moore Products Co. (Model No. 44). The overall flow control system is shown schematically in Figure 15. This system is similar to that shown in Figure 7,

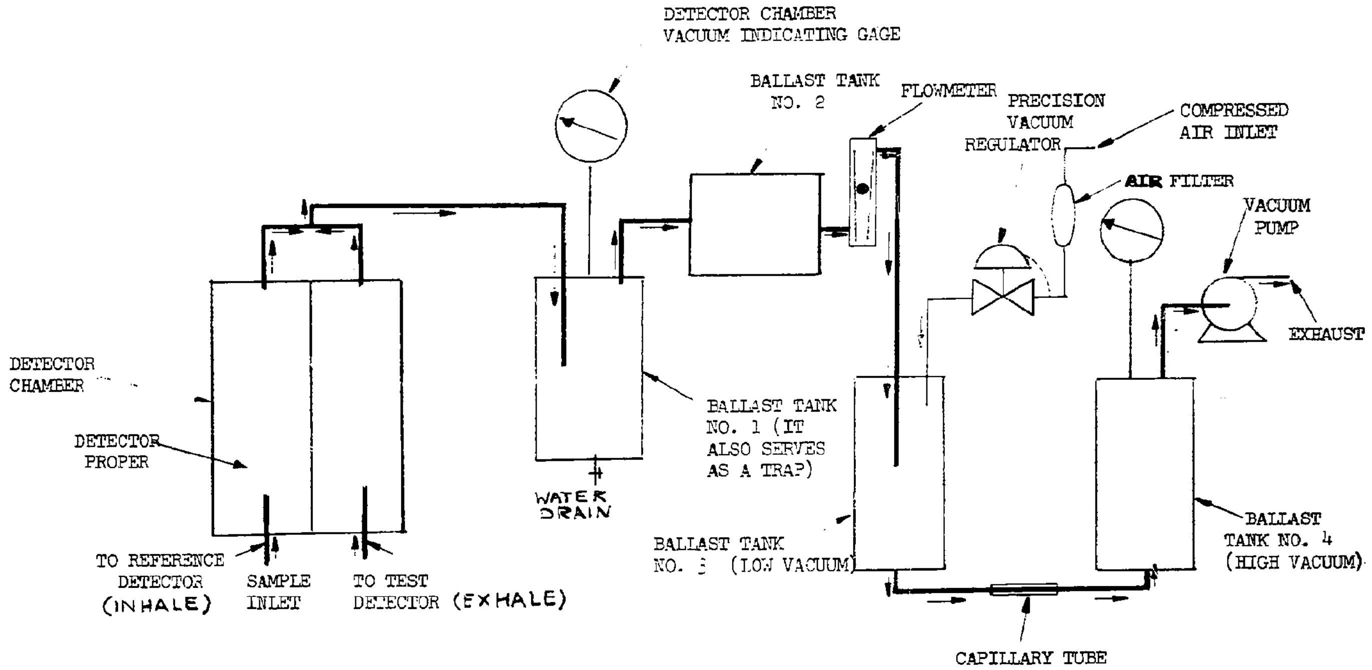

Figure 15 FLOW CONTROL SYSTEM FOR THE FINAL HFI DETECTOR

however, one more ballast tank was introduced, a capillary restriction was placed between the "high" and "low" vacuum ballast tanks, and most important, a precision vacuum regulator was added to the system. This vacuum regulator maintains "constant" vacuum in ballast tank No. 3. The vacuum within this tank is not really constant, but the variation in pressure is significantly reduced through the use of the upstream two ballast tanks. In actual operation, one may observe slight fluctuation of the flow as shown by the flowmeter. This fluctuation subsequently is "bucked out" and the indicating Magnehelic gage registers constant pressure.

The most important fact is that the problem of detector signal fluctuation has been virtually eliminated, and the detector became operable to its maximum sensitivity (few micrograms per liter range).

3.2.5 Bench Evaluation of the Final Model

This integrated detector was bench tested essentially in the same manner as the previous detectors. The signal output of the two detectors could be controlled through adjustment of air and hydrogen mixtures. Such adjustment is necessary to align the two detectors to yield the same signal when identical concentrations of the aerosol are being sampled. This adjustment can be also made by changing the position of electrodes within the detector, however, this latter adjustment should only be made after the detector was disassembled, cleaned, and reassembled again. Even at this point such an adjustment may not be necessary. Operation and service maintenance of this detector is presented in detail in the Appendix.

At the electrometer attenuation of 30X, detector chamber vacuum of 6" H_2O, 40% of full scale deflection of the recorder pen is obtained when aerosol simulant such as BSP in concentration of 2 micrograms per liter is taken into the bench test detector. These results were sufficiently indicative that integration of the components and the final assembly of the Inhalation Aerosol Dosimeter should be undertaken. These steps are discussed in the following sections.

SECTION 4

INTEGRATION OF COMPONENTS

This section presents the selection and evaluation of other basic components that are part of the Inhalation Aerosol Dosimeter.

4.1 Tidal Volume Measurement

The tidal volume represents the volume of air inspired and expired during normal or "quiet" breathing. This volume varies with the rate of breathing, and it also varies among individuals. Measurement of this volume per se is not essential in establishing the total dosage of the aerosol retained by the test subject, and this is especially true when the test subject is breathing the aerosolized air for any significant length of time, where the total volume of air inhaled is equal to that exhaled. However, to make the instrument more versatile it was provided with a sensor, digital readout and a recorder for the volume of air inhaled.

The sensor is of the hot-wire-anemometer-type, supplied by Hastings-Raydist Corp. of Hampton, Virginia. It is a modified version of their Model SM-20K, and it was developed for the purpose of measuring respiratory volumes. The filaments are kept at about 200°C, and according to the manufacturer, aerosol in the micron size particle range and in concentration range as stated in the contract should not have any significant effect upon the sensor.

The signal obtained from this sensor is fed into a Honeywell recorder which is equipped with a Disc integrator, pulse generator, and a separate counter. In this manner a record is made on the recorder chart of each individual volume of air inhaled, and the total cumulative volume of air inhaled is shown by the digital counter.

While the sensor response is not linear, it provides a very good approximation under normal breathing conditions. It may be necessary to calibrate this sensor for individual test subjects. A typical response of about one count per 30cc of air inhaled has been established for the instrument.

It may be noted that the Honeywell recorder had to be equipped with a variable resistor as the signal output from the sensor was considerably more than one millivolt.

4.2 Detector Signal Coordination

As mentioned previously, the Inhalation Aerosol Dosimeter is equipped with a dual-type HFI detector for measuring concentration of the aerosol in both the inhaled and exhaled streams. Since the detector signal is directly proportional to the concentration of aerosol in the sampled air stream, and since the two signals are essentially the same when the same aerosol concentration is being sampled. any deviation between the two signals must be directly proportional to the change in aerosol concentration in the two streams. If the signal output on the inhale stream is 0.90 millivolts and that on the exhale stream is 0.50 millivolts, the difference between these two signals (0.40 millivolts) is directly proportional to the change in concentration. That is, the higher the change in concentration in the two streams, the higher will be the signal difference. Note that the major assumption is that the respiratory volume of air exhaled is replaced by the same volume during the next inhalation phase. While this assumption is not true in general for a single respiratory cycle, the resulting deviations will cancel out and the assumption as to total volume approaches validity as the number of respiratory cycles increases. That is, the total volume of air exhaled must be equal to the total volume of air inhaled. Figure 16 shows diagramatically the

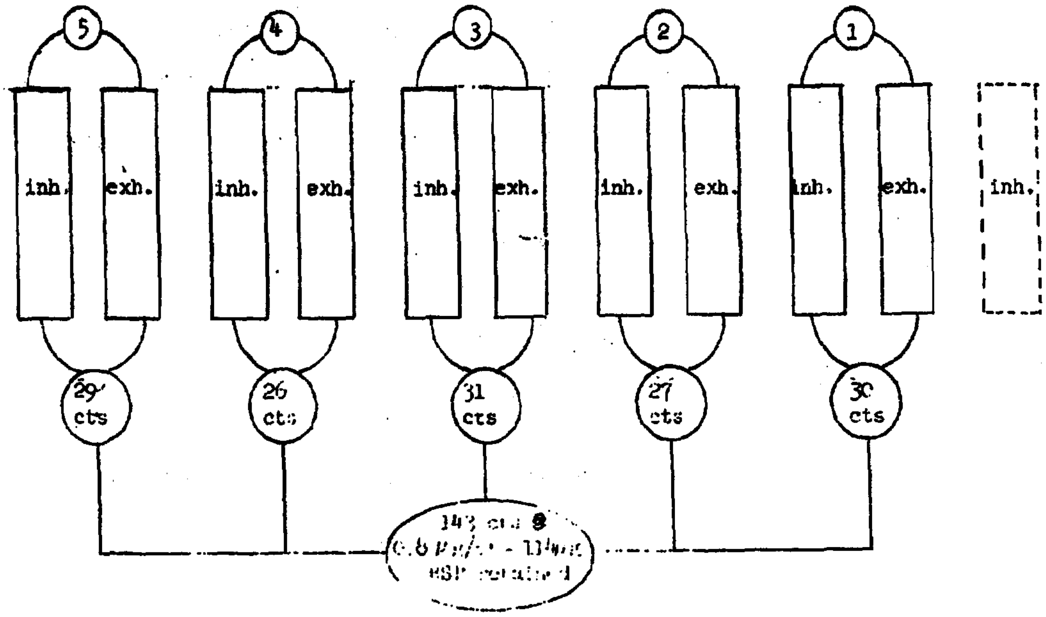

FIG. 15 DIAGRAMATIC REPRESENTATION OF THE DIGITAL READOUT IN THE INHALATION AEROSOL DOSIMETER

above sequence. If the exhaled and inhaled volumes are assumed to be the same, as shown in Figure 15, the signal difference, e.g. of 30 counts in respiratory cycle #1, represents the absolute amount of the aerosol withdrawn from the system, or the amount of the aerosol retained by the individual during the first respiratory cycle.

In the operation of the Inhalation Aerosol Dosimeter all three signals are recorded, so that visual pattern of each respiratory cycle is provided. Digital readout is provided for cumulative amounts of "agent inhaled" and "agent retained". There was no need to provide a digital readout for "agent exhaled" as the signal due to "agent exhaled" is electrically subtracted from the signal resulting from "agent inhaled" and the difference is shown by the digital readout of "agent retained".

The above operation perhaps can be explained in a simpler manner by referring to the recorders and observing the recorder pen position. For example, if the recorder pen on the "inhalation recorder" indicates 0.90 millivolts, and that of "exhalation recorder" indicates 0.70 millivolts, then the pen on the "agent retained recorder" must indicate 0.20 millivolts if the system were properly balanced prior to use. Figure 17 shows schematically the complete coordination of electrical signals in the Inhalation Aerosol Dosimeter.

4.2.1 Actuation of Counters

The "agent inhaled" and "agent retained" counters are actuated simultaneously by a microswitch located in the "air inhaled" recorder. This microswitch rides over a bakelite cam attached to the recorder pen mechanism, and set in such a position that when the "air inhaled" recorder pen moves from zero to about 0.05 millivolts, the other two counters are actuated. Note that this position can be changed by simply rotating and setting this bakelite cam.

The actuation of counters by the microswitch occurs right after the inhalation starts. The setting of 0.05 millivolts was selected so that essentially instantaneous instrument response could be obtained. A lower setting could result in false signal actuation as there may be some background noise coming from the tidal volume sensor. Thus the detector measures the aerosol concentration of the previously exhaled breath and the concentration of the aerosol in the inhaled stream. Note that the actuation of the counters is actually made through a mechanical link between the cam and a small wheel of the microswitch that rides over the cam. The initial actuator is the hot wire filament of the Hastings-Raydist Flowmeter, whose prime function is to measure the volume of air inhaled, and actuation of the counter is made as the "air inhaled" recorder pen moves upward.

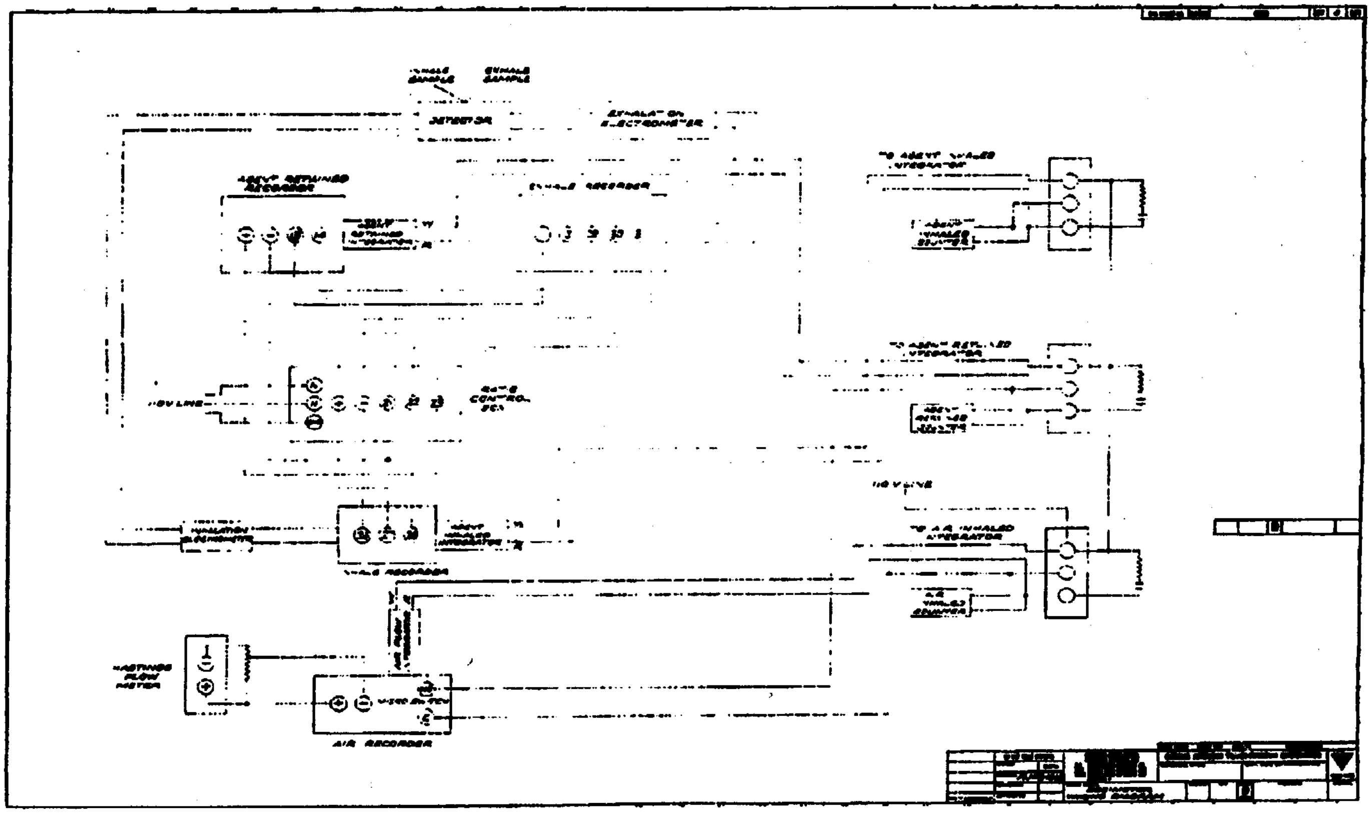

Figure 17 INHALATION AEROSOL DOSIMETER WIRING DIAGRAM

4.3 Detector Vacuum Control

Precise control of the detector chamber vacuum is extremely important as it is very closely related to the sampling rate of the aerosol. Figure 18 shows a complete flowchart of the sampled aerosol as it passes through the HFI detectors and through the exhaust system. A precision type vacuum regulator (Model No. 44, Moore Products Co.) is used in this system. The detector vacuum is read indirectly, as the vacuum gage (Magnehelic, 0 to 6" H_2O, F. W. Dwyer Mfg. Co.) is connected to the "moisture trap" (see Fig. 18), and since this moisture trap is linked with the detector through a 3/8-inch diameter Teflon tube, the pressure drop across this tube may be considered as negligible.

4.4 Detector Fuel Control

Both detectors use bottled hydrogen (chromotagraphic grade) and a mixture of oxygen and nitrogen. For best results of response and stability of the detector it was found experimentally that about 10% O_2 and 90% N_2 (both water pumped) should be used. This low proportion of oxygen to nitrogen is necessary because when ordinary water pumped air is used a signal results from the exhale detector even though there is no aerosol supplied to it. This signal apparently is due to the different composition of the exhaled breath. When 10% oxygen is used this problem is eliminated. Figure 18 also shows the linkage of the supply of fuel to the detector.

Supply of both the hydrogen and the O_2-N_2 mixture is controlled with a set of regulators and capillary tubes. These capillary tubes were adjusted in length so that approximately the same flowrate is obtained when the gas is fed at the same pressure.

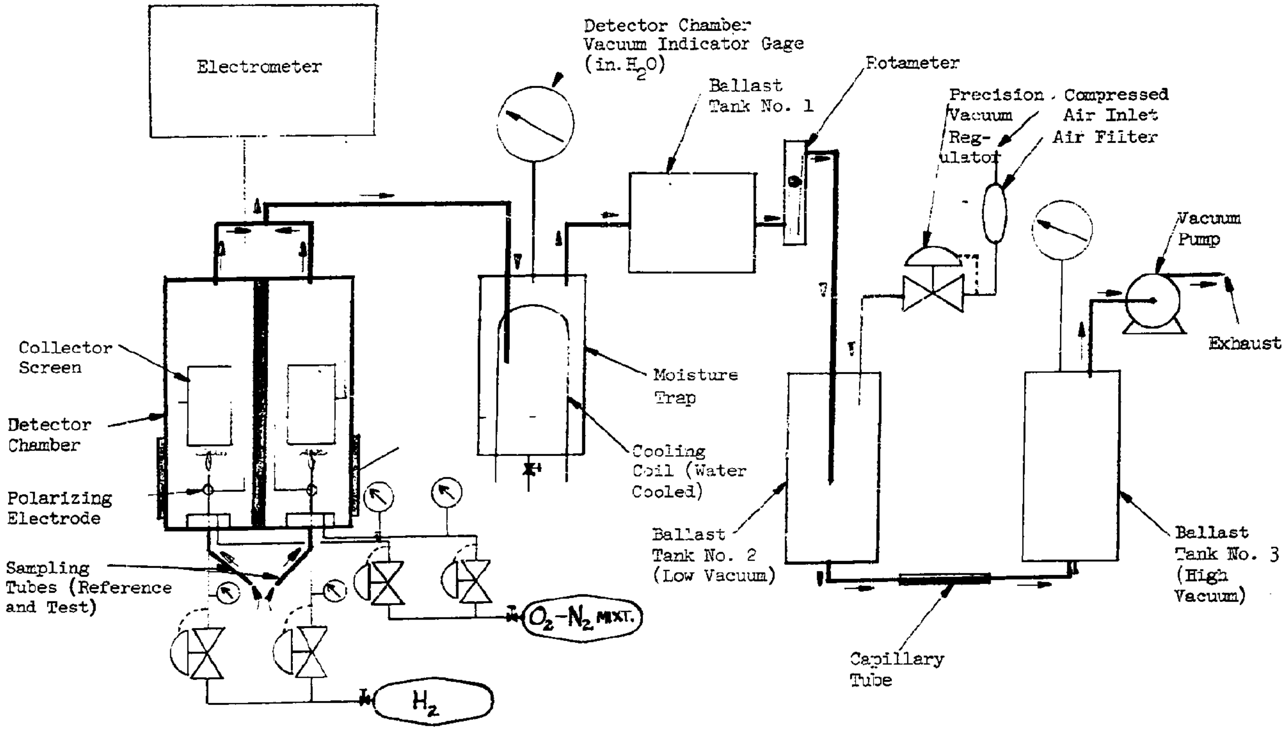

Figure 18 SCHEMATIC DIAGRAM OF THE FLOW OF SAMPLED AEROSOL THROUGH THE INHALATION AEROSOL DOSIMETER

The flow of hydrogen as a function of pressure is shown in Figure 19, and that of the O_2-N_2 mixture is shown in Figure 20. It is the hydrogen flowrate that is critical in the operation. At high hydrogen flowrates the background noise is too high, at low hydrogen flowrates the flame is extinguished. Best results are obtained at the hydrogen flowrate of about 10 to 15cc/min (4 to 5 psig) and the O_2-N_2 mixture of about 300cc/min (10 psig).

The capillary tubes supplying hydrogen and the O_2-N_2 mixture are equipped with a Millipore Filter Holder (Model No. XX3001200, Swinn Adapter) to filter out any particulates from the gas stream. Both the filter and prefilter should be replaced after about 1 year in service (See Appendix B, Maintenance Procedure).

4.5 Mask Assembly

The face mask used in the Inhalation Aerosol Dosimeter is a modified version of the mask supplied by Sierra Madre Engineering Co., Sierra Madre, California (Model No 450-MBU5P). The modification was made by reducing the length of tubing and shifting forward the inhalation-exhalation valve.

This mask was chosen for the IAD because of its small "dead volume" space, and especially because of the simple dual (inhalation exhalation) valve (Model No. 211-250) that is supplied with the mask which further reduces the "dead volume" space.

The valve is shown schematically in Figure 21, and its enclosure into the mask assembly is shown in Figure 22. The complete mask assembly is shown schematically in Figure 23.

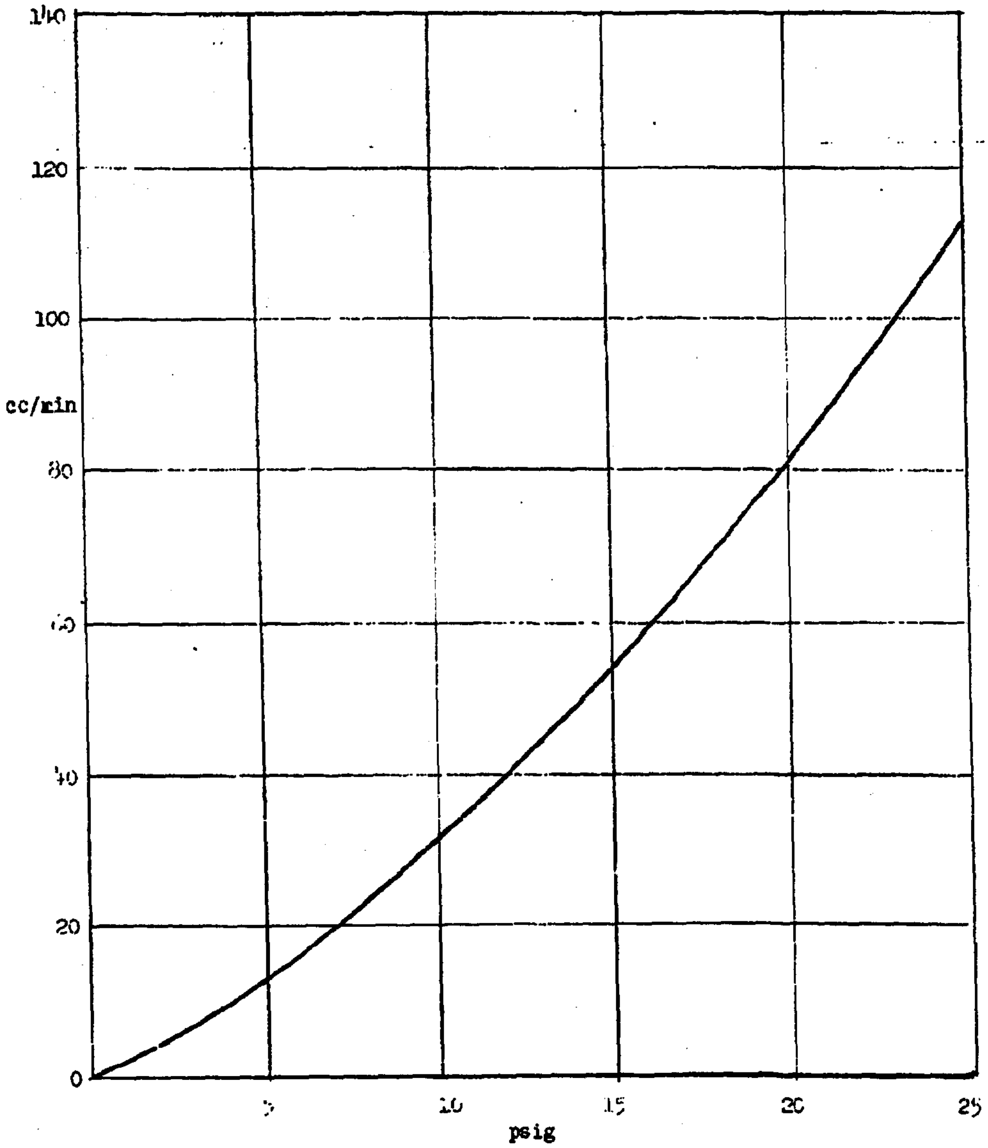

Figure 19 HYDROGEN FLOW RATE (cc/min) AS A FUNCTION OF REGULATOR PRESSURE(PSIG)

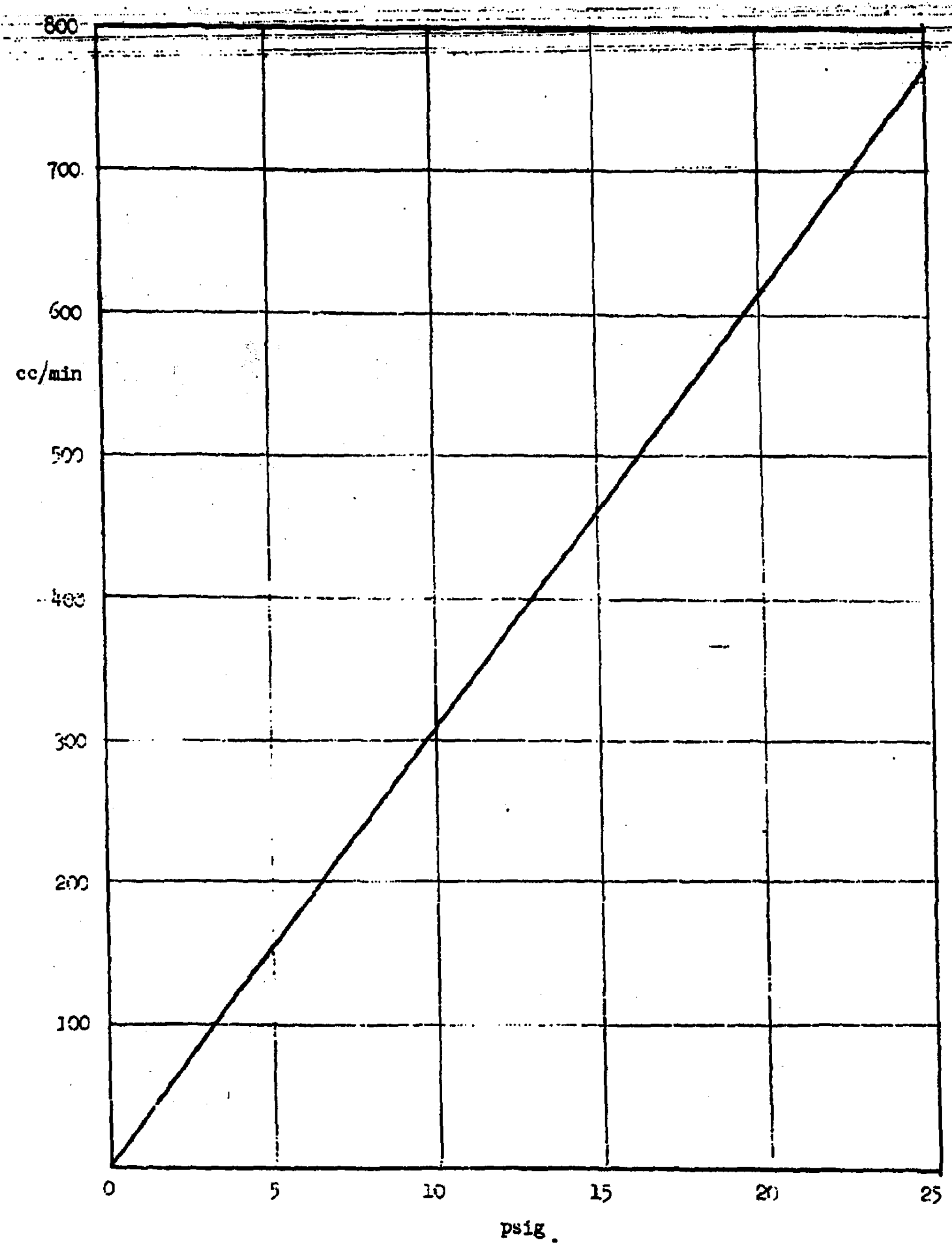

Figure 20 "PRIMARY AIR" FLOW RATE (cc/min) AS A FUNCTION OF REGULATED PRESSURE (psig)

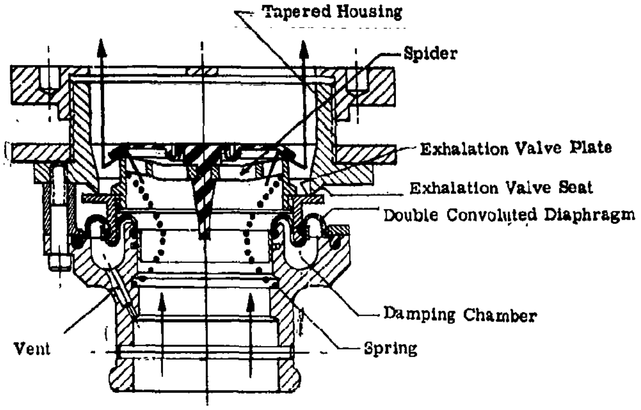

Figure 21 INHALATION-EXHALATION VALVE

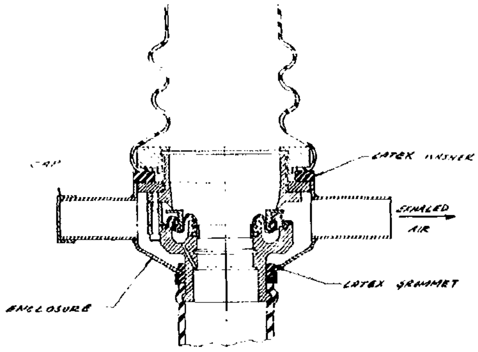

Figure 22 ENCLOSURE OF THE INHALATION-EXHALATION VALVE

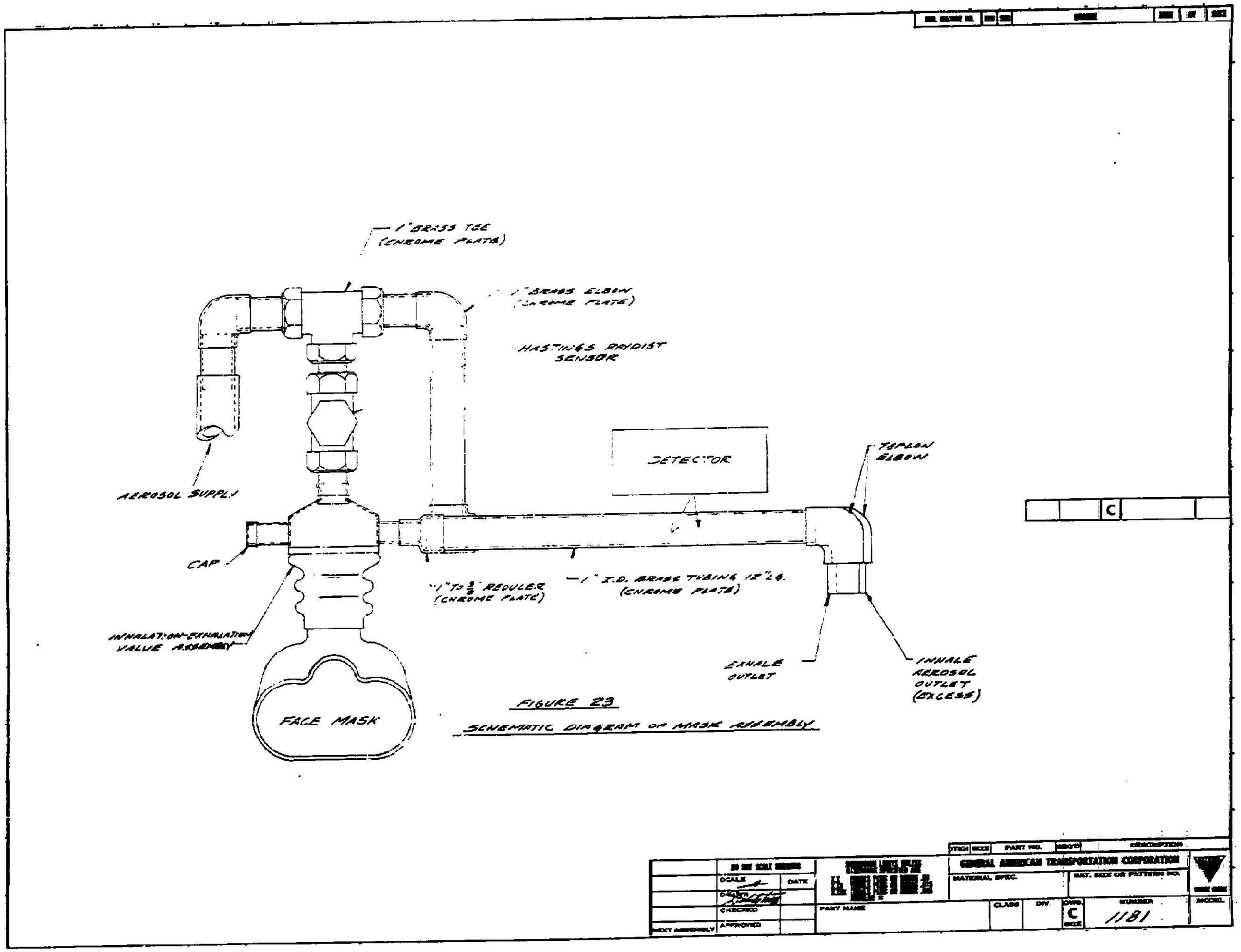

FIGURE 23

SCHEMATIC DIAGRAM OF MASK ASSEMBLY

4.6 Electrometers

The two electrometers used in the IAD were purchased from Research Specialties Co., Richmond, Calif. These electrometers were modified slightly; the modification made was in the flame igniter circuitry. Figure 24 shows the circuit diagram of the Model 605-3 Electrometer, as modified by MRD.

4.6.1 Electrical Specifications

Current Sensitivity:	1.25×10^{-12} ampere full scale response in most sensitive setting
Peak-to-peak noise level	(with detector cable connected): 2.5×10^{-14} ampere (2% of full scale in most sensitive setting).
Drift:	Less than 3% of full scale per degree Centigrade change in ambient temperature in most sensitive setting.
Line Voltage Stability:	Less than 0.5% of full scale change in output for ± 10% change in line voltage in most sensitive setting.
Attenuation Factors:	x1, x3, x10, x30 -- -- x 30 million in 16 steps.
Recommended Recording Meter:	0-5 millivolt span 1 second full scale response (output for other recorder sensitivities available on special order)
Electrometer Output Impedance:	700 ohms max - negative terminal grounded
Detector Polarizing Voltage:	300 volts (battery)
Maximum Background Suppression Current:	4.5×10^{-10} amp (45-volt battery used)
Detector Sensitivity:	5×10^{-13} moles/sec. (measured by exponential decay method using propane.
Power Requirements:	115 volts ±10%, 50/60 cycles, 200 watts

4.6.2 Detailed Description of Operation of the Electrometer

The electrometer circuit is operated by the AMPLIFIER rotary switch S3 and is fused by 1/2 ampere fuse F3. The electrometer circuit includes two batteries: the HV battery (B1 - 300 volt) which polarizes the detector for efficient ion collection and the background suppression or bucking voltage battery (B2 - 45 volt). The latter is used to supply a current through a 10^{11} ohm resistor R3 connected to the input of the electrometer which is adjusted to balance (or cancel) the background ionization current in the detector. The bucking voltage is adjustable from 0 to 45 volts by means of the COARSE and FINE BALANCE controls R1 and R2.

The AMPLIFIER rotary switch S3 has STANDBY and OPERATE positions. The former position energizes the electrometer amplifier but leaves the battery voltages disconnected. The OPERATE position adds the battery voltages so that the detector can be operated. The STANDBY position has been provided to permit turning on the electrometer circuit with no connected source of input current which might cause an initial surge of grid current in the electrometer stage resulting in a long recovery period. Furthermore the STANDBY position permits the electrometer to be left on in its warmed-up stable condition while the detector is unenergized during off periods for servicing and cleaning.

The electrometer is a d.c. amplifier with negative feedback. The input stage utilizes a CK5889 electrometer tetrode V1. The following two amplifier stages are transistors TR1 and TR2. The output voltage taken from the collector of the 3rd stage is connected to the input through one of a set of resistors selected by the ATTENUATOR switch S6A. Thus there is negative feedback of current to the input and the amplifier output voltage is linearly related to the ionization current. The current sensitivity is selected by the ATTENUATOR by virtue of the selection of feedback resistor.

A second wafer S6B on the ATTENUATOR switch is used to attenuate the output voltage connected to the recording meter. Thus the signal to the recorder is determined not only by the feedback resistor but also by the value of output voltage attenuation. The result is an overall attenuation range of 30 million to one obtained with five different values of feedback resistance. The advantages of output voltage attenuation are:

a) Amplifier drift and line voltage sensitivity are thereby attenuated.

b) The number of feedback resistors is reduced.

It should be noted that the two largest feedback resistors, R13 and R27, are 10^{11} and 10^{10} ohms, respectively, (differing by a factor of 10) and that only two output voltage attenuation settings are used in the x1, x3, x10, and x30 positions; whereas in the remaining positions, the feedback resistors R14, R15, and R16 differ by factors of 100, and four output voltage attenuation settings are utilized. The reason for this is that the response time of the electrometer-detector system depends on the value of feedback resistance. In the x1 and x3 positions, the response time constant is approximately 1.8 seconds. In the x10 and x30 positions the response time constant is 0.18 second because the feedback resistor is 10^{10} ohms instead of 10^{11} ohms. If the 10^{10} ohm resistor had been omitted, there would have to be four attenuator positions with the 1.8-second time constant. This would preclude taking advantage of the inherent rapid response of the detector in four rather than two attenuator positions. The response time of the circuit in attenuation settings higher than x30 is less than 0.18 second.

The statements in the last two paragraphs can be summarized as follows:

a) Electrometer drift if greatest in the attenuation settings x1, x10, x100, x10K, and x1M and less in all other settings by the factor of output voltage attenuation.

b) Response time is 1.8 seconds in the x1, and x3 settings and 0.18 second or less in all other settings.

There are two adjustments in the electrometer which set the amplifier output voltage to zero. (These adjustments are separate from the COARSE and FINE BALANCE controls which have to do with bucking current). A coarse adjustment can be made by varying the R7 potentiometer located on the rear of the Electrometer. A fine adjustment can be made on the front with the knob labeled AMPLIFIER ZERO. Both adjustments vary the screen voltage of the electrometer tetrode, thereby varying the plate current. These adjustments are used to set the amplifier output voltage to zero when there is zero input current (including zero bucking current). The coarse adjustment is set at the factory but may require occasional resetting as the electrometer tube ages.

The second stage transistor TR1 is a Silicon-type; a germanium-type thermistor is used for the third stage (TR2). This combination of transistor types results in minimum drift with ambient temperature changes.

The operating voltages for the electrometer circuit are supplied by two Zener diodes (D5 and D6) in series, which are in turn supplied by a regulated power supply. The series regulator element of this power supply is a 2N242 power transistor TR3. The regulator amplifier is a 2N377 transistor TR4.

The sensitivity of the electrometer circuit to line voltage changes is small. The major source of drift is ambient temperature variations (see specifications).

The Model 605-3 contains a flame ignitor circuit which consists of a filament transformer T4 whose 2.5-volt secondary leads are connected through the FLAME IGNITER push button switch S4 to the detector posts.
The 2.5-volt transformer is used to heat up a platinum igniter coil in the detector which results in the ignition of the hydrogen flame. Power is supplied to the primary of the igniter transformer T4 only when the amplifier switch is in STANDBY or OPERATE.

A Meter Zero Control is provided optionally for off-setting the recorder if so desired. The marker pushbutton cable plugs into the rear panel of the 605-3.

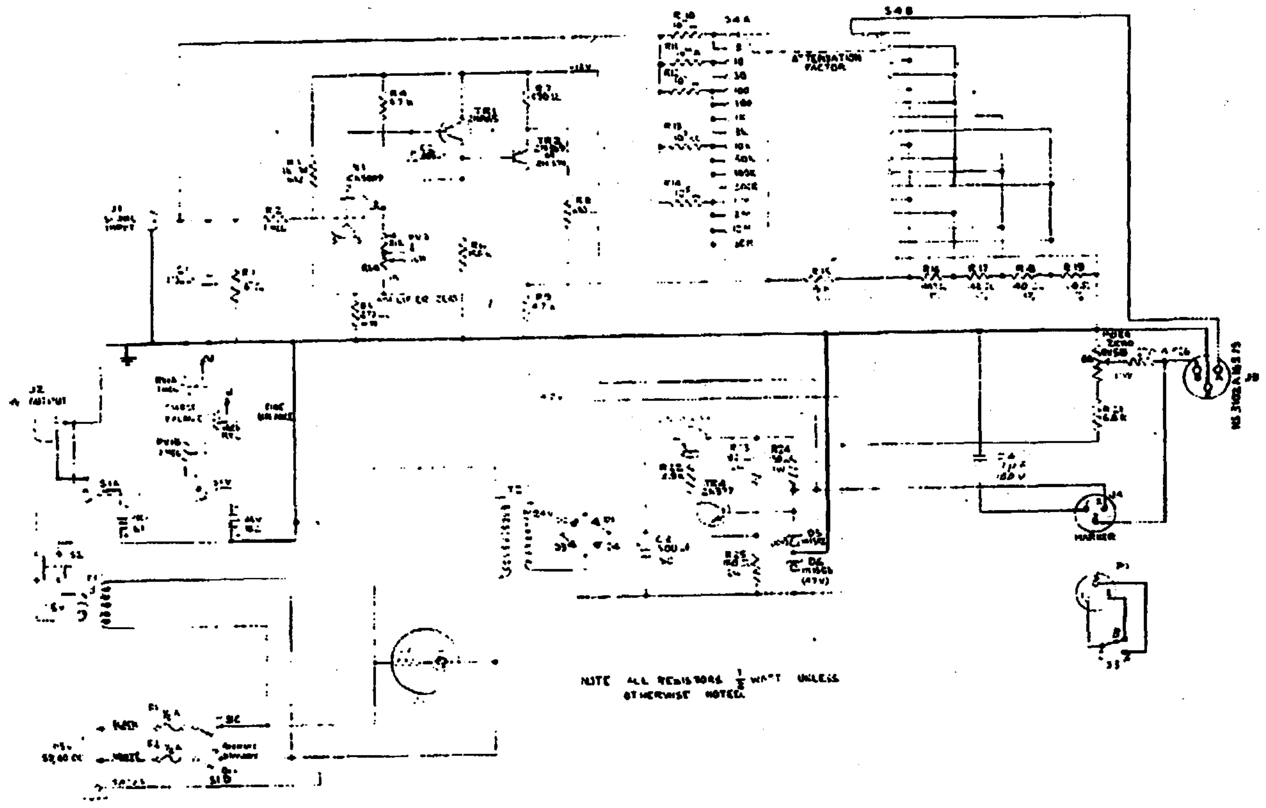

Figure 24 . CIRCUIT DIAGRAM OF ELECTROMETER MODEL 605-3

SECTION 5

ASSEMBLY AND EVALUATION OF THE INHALATION AEROSOL DOSIMETER (IAD)

5.1 Assembly

Standard type Emcor (Elgin Metalformers Corp., Elgin, Illinois) consoles are used to house all the components of IAD. The complete assembly, as shown in Figure 25 consists of three sections. The two side sections (FR-24A) primarily house the recorders; the section in the center houses the control panel and the other components.

Figures 26 through 29 give exact dimensions and positioning of the various components within the console assembly. Figure 30 is a closeup photograph of the control panel.

5.2 Evaluation of the Inhalation Aerosol Dosimeter

After the IAD was assembled at MRD, it was thoroughly tested for any malfunctions that could have occurred in the final assembly. Preliminary tests were carried out using BSP aerosol and live subjects in establishing the proper function of the instrument. Figure 31 shows a photograph of a subject placed on-stream and breathing BSP aerosol. Preliminary data indicated that retention of aerosol varied among individuals; when the concentration of about 80 mg per liter was breathed, the percentage retained varied from the low of about 35% to a high of 70%. Also, it was evident that percent retention varied inversely with the breathing rate.

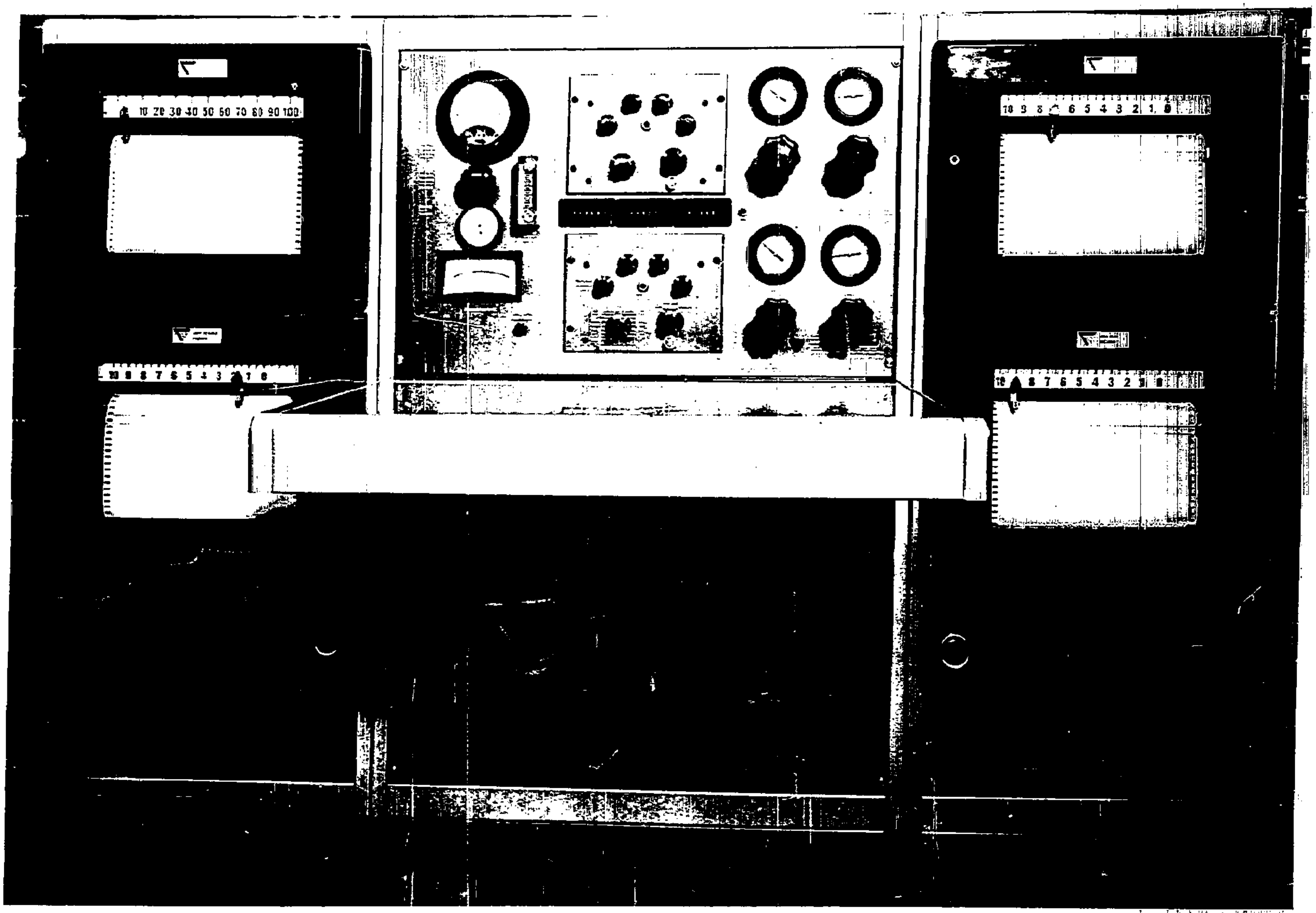

Figure 25 FRONT VIEW OF THE COMPLETELY ASSEMBLED
INHALATION AEROSOL DOSIMETER

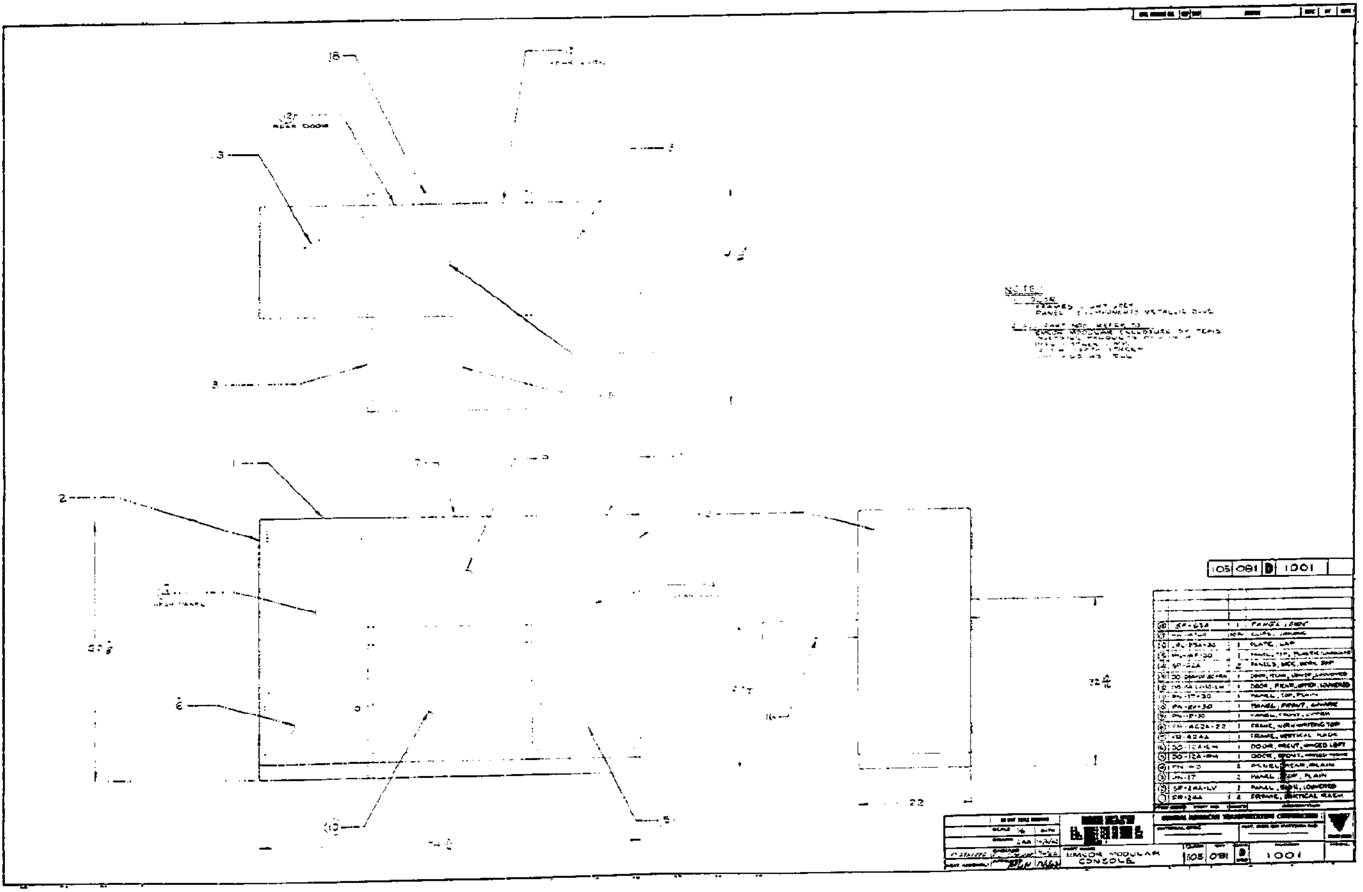

Figure A6 EMCOR MODULAR CONSOLE

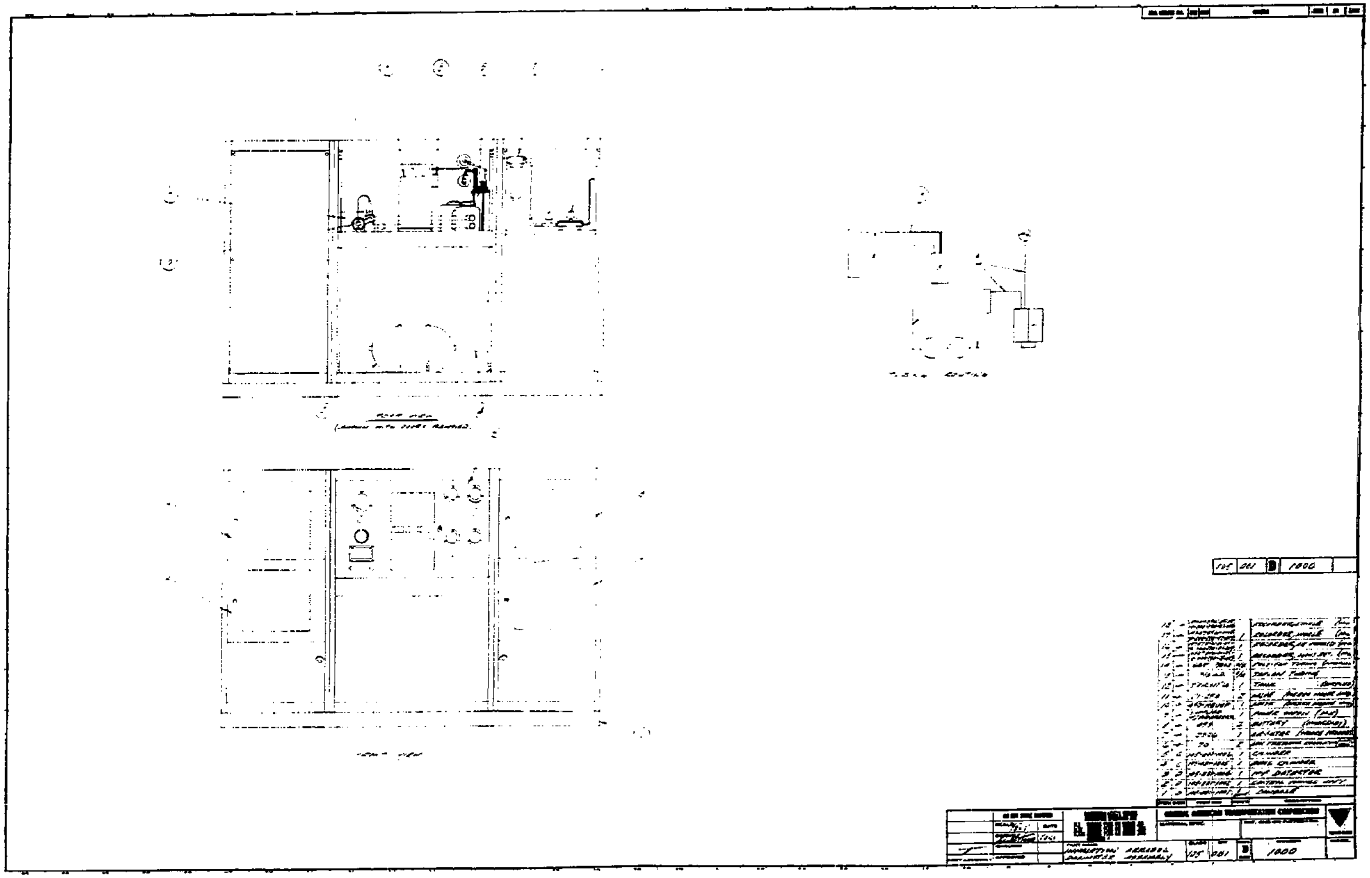

Figure 27 INHALATION AEROSOL DOSIMETER ASSEMBLY

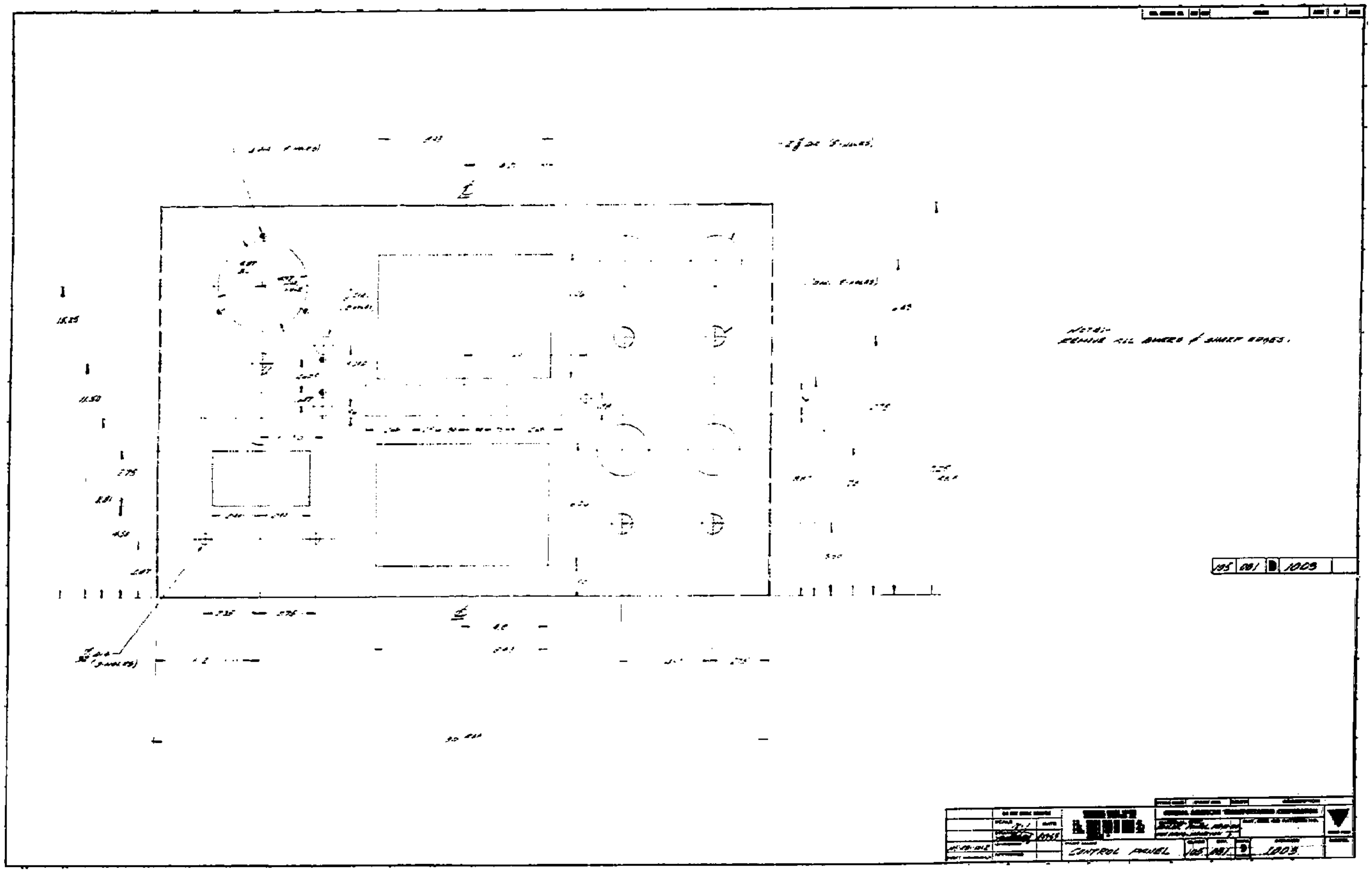

Figure 28 CONTROL PANEL DIMENTIONING

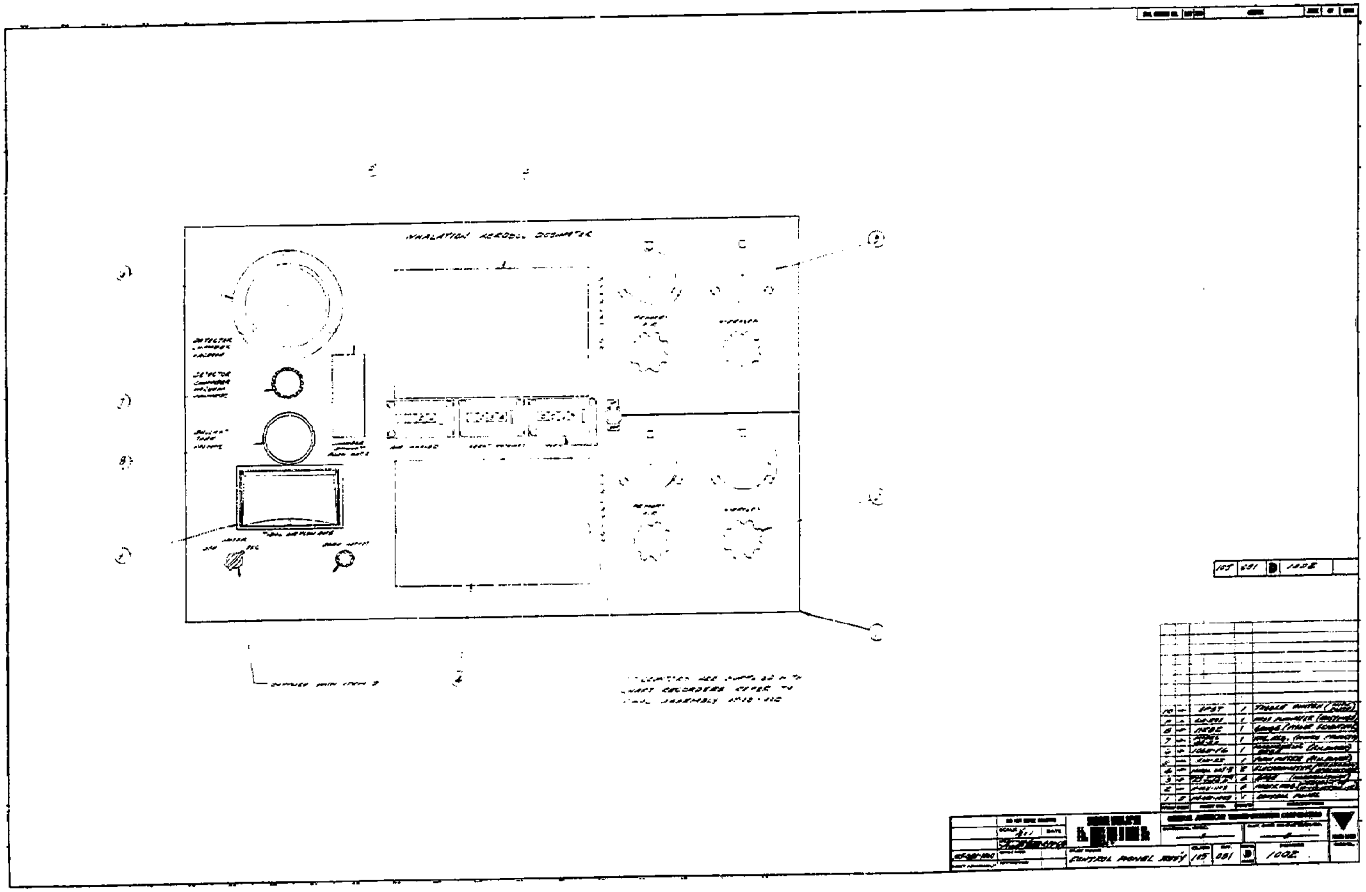

Figure 39 CONTROL PANEL ASSEMBLY

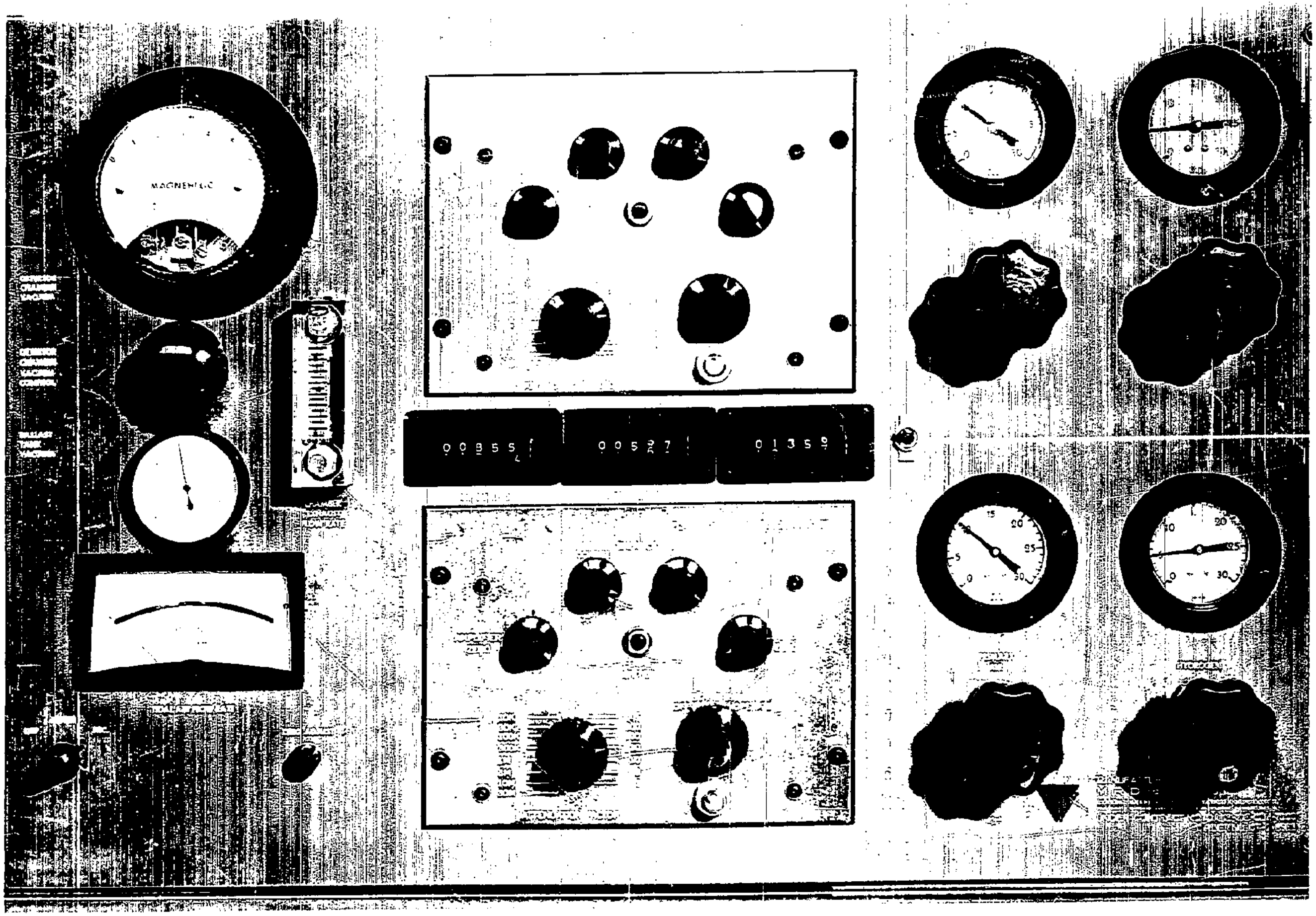

Figure 30 PHOTOGRAPH OF THE IAD CONTROL PANEL

Figure 31 PHOTOGRAPH OF THE SUBJECT INHALING BSP AEROSOL WHICH IS BEING ANALYZED AND MONITORED BY THE IAD SO THAT CUMULATIVE DOSEAGE OF THE AEROSOL RETAINED COULD BE ESTABLISHED

After the preliminary tests were completed, the unit was crated and shipped to CRDL for assembly and demonstration.

In operating the unit the initial problem appeared to be that of supplying proper mixture of oxygen and nitrogen, as it had to be fed from two separate tanks through a manifold arrangement. This problem was subsequently overcome with the aid of a paramagnetic oxygen analyzer (Beckman) so that the oxygen concentration fed into the detector could be maintained at about 10%. After this problem was solved, a total of seven tests were made with BSP aerosol, using two subjects.

Table 1 lists the data obtained. All the results shown in Table 1 were based upon previous chemical analysis of the BSP aerosol and correlating it with the signals obtained from the detectors. These chemical analyses were made by the standard colorimetric technique developed by CRDL. Table 2 lists the data showing signal response of the detector as a function of the chemical concentration of BSL aerosol in air (μg/liter). Figure 32 shows a graph of these data.

The Hastings-Raydist flowmeter (volume of air inhaled) was calibrated at CRDL with the aid of a spirometer. It was established that in the normal course of breathing the flow sensor actuates the counter at the rate of 33 counts per liter of air inhaled.

As Table 1 indicates, excellent reproducibility of the results was obtained for each individual subject. However, as was also observed at the MRD laboratory, there is significant difference in the percentage of BSP retained by the two subjects. This difference perhaps can be explained on the basis of breathing rate and volume, the lung size, and other physiological differences between the two individuals.

TABLE 1 DATA OBTAINED IN THE FINAL EVALUATION OF THE INHALATION AEROSOL DOSIMETER AT CRDL

Subject	Air Inhaled		BSP Inhaled		BSP Retained		Percent BSP	Attenuation 300X	
	Counts	Liters	Counts	μg	Counts	μg	Retained	m.v.	μg/lit.*
A	380	11.5	338	420	235	292	69.5	5.7	36.5
	346	10.5	349	430	230	284	66.0	6.3	41.0
	388	11.7	407	540	269	357	66.0	7.0	46.0
B	279	8.5	249	348	119	168	48.0	6.3	41.0
	300	9.1	306	310	130	131	42.4	5.4	34.0
	307	9.3	302	307	121	123	40.2	5.3	33.0
	338	10.0	281	320	121	138	43.1	5.2	32.0

* These data were obtained from a calibration curve estalished on the basis of actual chemical analysis and the signal response of the detector (See Figure 32).

TABLE 2 CALIBRATION OF DETECTOR AT ATTENUATION OF 300X

Signal Output, m.v.	μg/liter*
2.2	11
4.5	30
6.0	39
9.0	60

* These analyses were made according to the standard procedure established at CRDL and with the instrumentation available at CRDL.

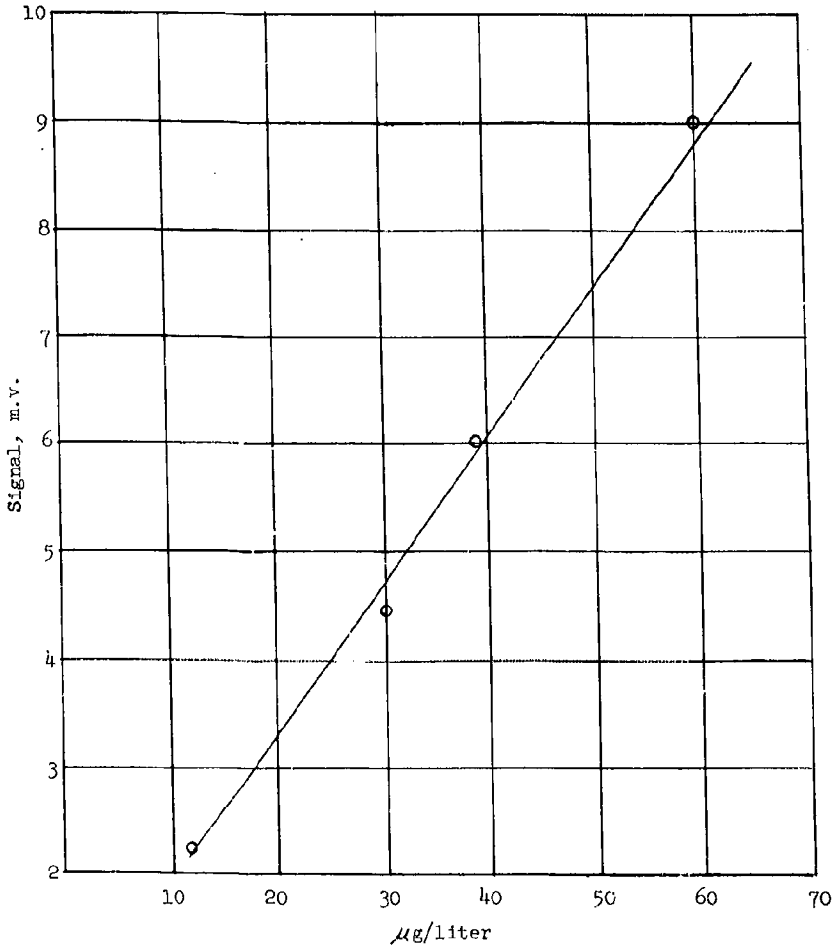

Figure 32 DETECTOR SIGNAL RESPONSE AT THE ELECTROMETER ATTENUATION OF 300X AS A FUNCTION OF BSP CONCENTRATION

SECTION 6

CONCLUSIONS

An Inhalation Aerosol Dosimeter (IAD) that meets all the requirements set out in the contract (No. DA-18-108-CML-7006) has been developed by the MRD Division of General American Transportation Corporation.

The overall construction of the IAD parallels the design suggested in the original proposal. The major modification of that design is that hydrogen flame ionization principle is used for detection and monitoring of the aerosol concentration, and the secondary modification consists of developing a very precise aerosol sampling system. The sampling system is based upon maintaining precise (reduced) pressure within the detector so that actual sampling is a truly continuous process.

SECTION 7

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APPENDIX A

OPERATING PROCEDURE

A. Operating Procedure

There are certain expendable materials that are required for the operating of the IAD that are not supplied. These required materials are listed below:

A.1 Required Materials

A.1.1 Gases

(a) Hydrogen - preferably chromotagraphic grade.

(b) Oxygen - Nitrogen Mixture - 10% oxygen - all in one cylinder. This mixture should be "water-pumped" type, that is, there should be no traces of hydrocarbons in it. This gas is ordinarily supplied in the Type IA cylinder (235 cubic feet). The cylinder should be equipped with laboratory-type regulators with standard type gages. The lower pressure range should be about 0-100 psig.

(c) Compressed House Air - this is ordinary type compressed air that is normally available in laboratories.

A.1.2 Water

Ordinary tap water to be used as the cooling fluid. This water is passed through the unit in essentially the same manner that one does in laboratory-type water cooled condensers. The flow rate should be about 100 ml per minute.

A.1.3 Power

Single phase, 105-125 volt, 60-cycle electric power. Less than 10 amperes are required.

A.2 Starting Procedure

The starting procedure is very critical and it MUST be followed very closely; this is very important as some of the delicate instruments (especially the Magnehelic gage) can be damaged if the procedure is not followed.

To start the instrument, proceed as follows, STEP BY STEP:

STEP 1. (a) Plug in the main electric cord and turn the detector heater to "on" about 2 hrs in advance. The switch is on the variable transformer located in the rear of the unit. The transformer should be set at about 25.

(b) Connect both hydrogen and oxygen-nitrogen mixture (primary air). These connections are made on the left hand side of the unit, facing the control panel. Both hydrogen and primary air should be supplied to the IAD at about 30 psig.
At this point neither hydrogen nor the primary air should indicate positive pressure on the control panel, (the gases should not flow into the detector). Their flow is controlled by the large knobs on the panel, and the knobs should be turned all the way left so that the gages read zero.

(c) Connect the cooling tap water and adjust its flow to about 100 ml per minute. This flow rate is not critical and generally "small dripping" is sufficient, especially when the water is less than 70°F; . its purpose is to condense the water vapor coming from the detector as a product of combustion.

(d) Turn both electrometers to STANDBY and all four recorders to "on". The switches are located in back of the recorders. Note that there is a main switch located at the rear of the IAD. This switch controls the power supply to electrometers and recorders. This switch should be used whenever any prolonged shut-down of the unit is made. At this point it is not necessary to turn on the chart speed of the recorders.

STEP 2. Step 2 and Step 3 should be done very quickly, therefore read the footnote below* to be familiarized with the principles involved, and then go ahead. Turn the diaphram pump to "on". Both the pump and the switch are located behind and underneath the control panel. It is accessible from the rear of IAD. Wait a short time (few seconds) until the Magnehelic gage (DETECTOR CHAMBER VACUUM) registers about 4 inches H_2O, then proceed to step 3.

STEP 3. Connect the "house air" to the IAD. The air should be supplied at about 30 psig, as it is further regulated to about 10 psig by a regulator located in the rear of IAD before it is delivered to the precision vacuum regulator (Model 44, Moore Products) whose control knob is located on the front panel and labeled DETECTOR CHAMBER

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* Steps 2 and 3 are very critical in the starting procedure. As the diaphram pump is turned "on" it immediately starts pulling high vacuum as shown by the BALLAST TANK VACUUM gage on the control panel. Subsequently, vacuum is being created in the other ballast tanks that are linked to the detector. Unless this vacuum is relieved (which is explained in Step 3) the Magnehelic gage can be damaged. This vacuum is controlled by the DETECTOR CHAMBER VACUUM CONTROL (DCVC) knob located on the front panel.

VACUUM CONTROL (DCVC). Observe the movement of the needle of the Magnehelic gage. If it moves rapidly to the left, immediately cut off the supply of "house air". Turn the DCVC knob slightly counterclockwise and repeat the above procedure, until a slight fluctuating movement of the needle to the right is observed. As the needle moves to about 5 on the scale, turn gently to DCVC knob clockwise, until the movement of the needle is arrested. Subsequently set the DETECTOR CHAMBER VACUUM with the DCVC knob to read about 4 inches water by very gentle turning of the above knob either clockwise or counterclockwise.

STEP 4. This step deals with the flame ignition of both detectors (inhalation and exhalation).

(a) Set the gas pressures as follows:

Hydrogen: 15 psig to ignite, 5 psig to operate.
Primary Air: 10 psig to ignite, same to operate.
Magnehelic gage: 4 inches of H_2O to ignite, 6 inches H_2O to operate.

(b) After the hydrogen has been flowing for a few minutes, the lines will be purged of air and the flame can be ignited. Push both FLAME IGNITER buttons simultaneously for about 1/2 to 1 second. Flame ignition is signified by a "dull" audible "pop" in the detector, also a quick temporary movement of the needle of the Magnehelic gage will be observed (to the left).

(c) Turn the amplifier switch to OPERATE position and adjust recorder reading with balance controls to approximately 10% above zero with attenuator in X100 position and allow the amplifier to stabilize. This stabilizing process may take some time if the electrometers were just turned on. Also the sampled air must be free of aerosol and/or any other organic contaminants.

(d) Remove the face mask from the mask assembly, attach the plastic diaphram bellows (as supplied) and pump it several times. The air normally goes into the inhale tube of the mask assembly and the purpose of using bellows is to bring in the same clean air into the exhale tube so that zeroing of both recorders could be done.

(e) Gently reduce the Hydrogen pressure to about 5 psig on both detectors and set the DCVC to indicate 6 inches H_2O vacuum. Turn the amplifier switch to STANDBY and the attenuator switch to one position counterclockwise beyond the 300M position. This is a position in which recorder zero can be set. Using METER ZERO knob, set recorder to zero position.

(f) Turn the attenuator switch to 1M position and adjust AMPLIFIER ZERO knob until recorder again reads at zero position. Turn the amplifier switch to OPERATE. Turn the balance control knobs (both coarse and fine) all the way clockwise. Turn the attenuator switch step by step clockwise. If the flames were ignited, the recorder pens will move upscale by the time attenuator factor approaches X10 or lower. When the flames are not lit, increase both hydrogen pressure to 15 psig, depress the FLAME IGNITER buttons (1/2 to 1 second) and observe the movement of the recorder pens. If the flame is ignited, the recorder pens will move and stay upscale. Proceed to (g) below. It is important to ignite both flames simultaneously. If the flame in one detector is ignited and an attempt is made to ignite the second flame separately this generally will result in extinguishing the other flame because of the rapid buildup in pressure when ignition takes place.

When the flames cannot be ignited, investigate as outlined in Appendix B, Trouble Shooting.

(g) If flames are not ignited, repeat (e) and (f). Turn the attenuator switch to X100. Set FINE BALANCE to its mid-point and turn course balance to zero the recorder approximately. (If this cannot be done, it indicates that either sampled air is impure or the amplifier is not warmed up). Use minor adjustments of FINE BALANCE for final zeroing of the recorders. The final zeroing can just as well be done at X10 or X1, but during the initial warmup and/or the pressure of contaminants in the air will either make the baseline drifting rapidly or excessive background noise will result from these impurities*.

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* At this time it will be useful to familiarize with the effect of the ionization current on the igniting and extinguishing of the detector flames. To do this, let the hydrogen pressure fall to zero. The flame will extinguish at about 2 or 3 psig. Now turn balance controls fully clockwise and zero amplifier if necessary. Turn hydrogen pressure to 15 psig and repeat flame ignition. When the flames ignite, both recorders will move upscale. Either X100 or 1K attenuator settings may be necessary to balance controls to zero the recorder again. Another very good check as to whether the detector flames are lit is to bring a small sample of organic vapor (or cigarette smoke) into the mask assembly-the pens will move rapidly upscale when the flames are lit, and it may be necessary to set the attenuator switch at 1K or 3K to keep the pens on scale.

STEP 5. Now that the instrument is zeroed, the next step is to coordinate the detector signal response. It may be necessary to make some adjustments to obtain the same signal response where the same concentration of the aerosol are brought into both tubes of the mask assembly*.

(a) Turn the attenuator switch to position X300 (this setting depends upon the concentration of the aerosol to be used in the dosimetry procedure; either lower or higher settings can be used. Supply aerosol into the mask assembly continuously at the rate of about 30 liters per minute. Depending upon the aerosol concentration, the inhale recorder pen will move up and indicate a certain signal output. Continue supplying the aerosol until this signal output shows no drift (this indicates that aerosol concentration is not changing).

(b) Pump the diaphram bellows several times and observe the signal output on the exhale recorder. Continue pumping until there is no change in signal output. If the signal output is the same on both recorders, the dosimetry can be started. If the signals differ, make adjustment as described below:

Signal coordination on both detectors can be made through

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* By identical construction of the detectors the sampling rate in each detector of the aerosol by both detectors was established to be the same when the detector chamber pressure (vacuum) is maintained constant. However, as indicated in discussing the theory of operation of HFl detector, the flow of current across the flame is directly related to the flow of hydrogen, and therefore, any difference in flow of hydrogen will cause a difference in the two signals.

either increasing or decreasing the flow of hydrogen. If the inhale recorder shows 0.80 millivolts and the exhale recorder shows 0.60 millivolts, the adjustment is made by either reducing hydrogen pressure to the inhale detector or by increasing it to the exhale detector.

Increase the exhale signal to about 0.85 millivolts by gently increasing the hydrogen pressure to this detector. Such an adjustment in general will require rezeroing of the electrometers. Cut-off the aerosol supply to the mask assembly, purge it with clean air and rezero the detectors as follows: Turn the attenuator switch counterclockwise one step past 30M, turn power supply to STANDBY, zero the recorders with METER ZERO knob, turn the attenuator switch to 1M and zero recorders with the AMPLIFIER ZERO knob. Turn attenuator switch to X300 and zero recorders with the BALANCE control knobs. Repeat (i) at the X300 setting and examine detector signal outputs. It may be necessary to make such an adjustment several times until essentially the same signal output from both detectors is obtained.

STEP 6. Analyze the aerosol content in the air stream colorimetrically or by any other method that would indicate absolute concentration of the aerosol. This step is necessary to calibrate the detector so that actual concentration of the aerosol can be correlated to the signal output. For example, as shown in Figure 32 of the Final Report, when BSP aerosol was used in the concentration of 50 micrograms per liter, the detector signal output at the electrometer attenuation of X300 was

0.75 millivolts, and it appears to be linear throughout the entire range. This indicates that one or two spot checks of the signal output versus actual aerosol concentration will be sufficient to calibrate the detector.

STEP 7. (a) Examine the setting of the signal actuating microswitch located in the inhale recorder. It should actuate the signals (both inhale and exhale) at about 0.05 millivolts. This setting is made through rotating and tightening the bakelite cam in back of the recorder-pen mechanism.

(b) Turn the air flow meter to REC. Adjust, if necessary, the recorder pen with the ZERO ADJUST screw, (the aerosolized air should be supplied into the mask assembly at about 30 liters per minute).

(c) Turn the counters switch to "on" position. Reset all three counters to zero by depressing the reset levers.

(d) Tell the test subject to put on the face mask and hold the breath temporarily (a second or less). Depress the reset lever on the AGENT RETAINED counter and while holding it down give a signal to the test subject to start inhalation. At the end of the inhalation phase release this lever and let the test subject continue breathing.

STEP 8. Now that inhalation is in progress observe the digital readouts. It may be necessary to make some arithmetic calculations to establish the actual dosage retained. The actual dosage of agent retained is established as follows (see Table 1 in Final Report):

The counts on AIR INHALED readout should be divided by 33 (33 counts per liter air, it varies slightly from subject to subject) to obtain volume of air inhaled (liters). Multiply this figure by the aerosol concentration which was established in STEP 6. This gives total micrograms of aerosol inhaled. Divide the total digital readout of AGENT INHALED by this number. This gives the detector response in terms of "counts per microgram". (This factor may vary, however, it is generally around 0.80). Multiply the digital readout of AGENT RETAINED by the above factor to obtain the total amount of agent retained in terms of micrograms.

After one is well familiarized with the operation of the IAD and the test subjects, all that will be necessary is to multiply the AGENT RETAINED by the previously determined factor to obtain the actual dosage in terms of micrograms of the agent administered.

APPENDIX B

MAINTENANCE AND TROUBLE-SHOOTING PROCEDURE

B. Maintenance and Trouble-Shooting Procedure

Section B.1 below describes general maintenance procedure of the IAD, and Section B.2 provides a general guide in locating and correcting "malfunctions" of the instrument that may occasionally occur.

B.1 Maintenance Procedure of IAD

The overall maintenance procedure of the IAD can be related to the maintenance of the basic components that comprise IAD. These basic components are the following:

(1) Recorders
(2) Electrometers
(3) Vacuum Control Assembly
(4) Hydrogen Flame-Ionization Detector
(5) Mask Assembly
(6) Tidal Air Flow Meter

The maintenance of these individual components is described below.

B.1.1 Recorders - there are four recorders in the IAD. These are high speed "Brown Electronik" recorders supplied by Honeywell. Their routine maintenance is listed in the table below. Any specific questions on the recorders as related to their operation and maintenance are referred to the manufacturer's manual.

ROUTINE MAINTENANCE OF RECORDERS

What to Do	When
Change Chart	As required
Refill ink reservoir	As required
Amplifier	
Check tubes	Once a month
Adjust sensitivity	Once a month
Replace fuse	As required
Slidewire	
Clean	Once a month
Replace contactor	As required
Clean scale	As required
Lubricate	As required

B.1.2 Electrometers - there are two electrometers, Model 605-3, supplied by Research Specialties Co., Richmond, California. Routine maintenance of the electrometers is required as follows:

a) Battery replacement:

Replace 300 volt battery once each year. Replace 45 volt battery once each year or whenever it appears to be putting out inadequate voltage. Replace batteries whenever they appear to be leaking their contents.

CAUTION: Use care in touching components inside the electrometer. The 300 volt battery can cause a serious electrical shock.

b) Electrometer tetrode type CK 5889:

This tube can be expected to last from 1 to 5 years. If replacement is necessary be sure not to touch any of the high impedance components in the electrometer circuit with anything but clean tools--never with the fingers. Clean components are an essential factor in the proper operation of this circuit. (The circuitry is shown in Figure 24.)

B.1.3 Hydrogen Flame Ionization Detector (HFID) - routine maintenance is required on the following components:

a) Detector burner tip may require cleaning if noise level increases inoridinately. The entire base, including all inlet tubes may also require cleaning. Clean with appropriate solvents and bake out while passing nitrogen through all tubes at 300°C for 1/2 to 2 hours.

b) The Teflon block supporting the detector electrodes should be cleaned periodically. This can be accomplished with a fine camel brush and using methonol as solvent.

c) The Millipore filters within the Swinny Adapter (attached to the capillary tubes supplying gases to the detector) should be replaced about once a year.

B.1.4 Tidal Air Flow Meter - this is a mass flowmeter, Model SM-20X, modified, 0-20,000 CC/min. range, with a Type K-28 flow tube (sensor) as supplied by Hasting-Raydist Inc., Hampton, Virginia. No routine maintenance is required for this instrument. The flow tube is discussed with the Mask Assembly.

B.1.5 Mask Assembly - consists of the face mask, dual inhalation exhalation valve, tidal air flow sensor, and brass (chrome plated) tubing. The complete mask assembly should be periodically disassembled and cleaned. The air flow sensor should be handled with extreme care - especially the sensing filaments should not be touched. It may be possible that over a period of time a thin film may deposit over these filaments, which in turn will lead to a decrease in sensitivity. This sensor should be calibrated periodically, and when the sensitivity drops significantly, the sensor whould be replaced (flow tube Type K-28, Hastings-Raydist, Inc., Hampton, Va.)

The other parts of the mask assembly should be washed with either plain water or with appropriate solvent to remove any of the deposited aerosol. Such cleaning should be done as required, and at any time prior to the use of different aerosol.

B.1.6 Vacuum Control Assembly - consists of a series of ballast tanks, a regulator and a filter. It is essential to keep this assembly free of foreign particles, oil, and water, to prevent clogging of the capillary restriction between the two ballast tanks, and also clogging of the restriction and pilot nozzle within the precision regulator (Model 44, Moore Products Co.). The restriction screw may be readily removed for cleaning by first removing the restriction sealing screw in the center casting and then the restriction assembly. A set screw wrench is provided in the base of the casting.

The knurled cleaning wire assembly, also mounted in the base of the casting, should be run through the restriction screw several times. After cleaning, the restrictions screw must be turned in tightly and its sealing screw replaced to restore normal operation. Such cleaning operation may have to be done once a year in normal operation.

The filter (Part No. 2306, Moore Products) entraps oil fog from instrument supply-air; it should be drained periodically (as necessary).

It may be necessary to check the Magnehelic gage periodically against a water manometer. If the gage can not be adjusted to zero and if its calibration has changed, it should be replaced.

B.2 Trouble Shooting

The following list of troubles and probable causes will serve as a guide in correcting malfunctions that may arise.

B.2.1 Detector Chamber Vacuum Control

a. Trouble - detector vacuum control cannot be established and/or maintained.

Possible cause -

1. Loose connections of tubing within the vacuum control assembly. Make sure that all tubes are tightened to make an air tight seal.
2. Regulator knob (DETECTOR CHAMBER VACUUM CONTROL) was turned to far in either direction. Follow

Steps 1 through 3 carefully as described in Section A.2, Starting Procedure.

3. Moisture collects in the flowmeter tube on the control panel. Make sure there is circulation of cold water through the "moisture trap". You may clean out the flowmeter tube with a pipe cleaner.

B.2.2 Electrometer and/or Detector Malfunction

a. Trouble - flame does not ignite

Possible cause -

1. Flame igniter circuit inoperative, (be sure attenuator factor switch is in X10 or lower positions when pushing flame igniter button, and the power switch in OPERATE). Check for electrical continuity between the two igniter posts in the detector. If there is no continuity, the igniter filament is burned out - the electrode assembly must be removed and a new filament (platinum wire 36 ga. about 1-1/2 inch long) should be silver soldered to the igniter posts.
2. Hydrogen line may be plugged or hydrogen may be supplied at too low a pressure. Also excessive flow of "primary air" may prevent ignition of the flame. At least 12 psig hydrogen pressure generally is necessary to ignite the flame.

b. Trouble - no recorder response obtained when amplifier is turned to STANDBY.

Possible Cause -

1. Recorder not operating.
2. Amplifier fuse F3 burned out.
3. Power Supply burned out. Check voltages in power supply.

c. Trouble - Recorder cannot be zeroed.

Possible Cause -

1. Amplifier required warming up.
2. Coarse zero adjusting needs resetting.
3. Power supply faulty. Check for proper voltage in power supply. (See Figure 24)
4. Shorted wiring in electrometer stage.

d. Trouble - Recorder responds to amplifier zero adjustment but does not respond when flame ignited.

Possible Cause -

1. Detector signal cable disconnected from electrometer.

e. Trouble - Excessive noise level from detector.

Possible Cause -

1. Room air sampled is contaminated either with the aerosol or some other organic vapors.
2. Dirty detector or gas lines.
3. Dirty detector insulators - clean the electrode supporting Teflon disk with camel hair brush and methanol. Clean especially around the anode (collector)post

4. Loose wiring at detector - check noise level of electrometer alone by disconnecting detector signal cable.

f. Trouble - Excessive noise level of electrometer with detector cable disconnected.

Possible Cause -

1. Line voltage transient.
2. Electrometer input components dirty or moist.

g. Trouble - Electrometer zero varies between attenuation settings of X1M and X1 with detector signal cable disconnected.

Possible Cause -

1. Electrometer grid current excessive. Try operating for 12 hours in STANDBY position. Then recheck.
2. Electrometer input components dirty or moist.

B.2.3 Signal Coordination and Control of Counters

a. Trouble - Counters (all three) are actuated when the counter switch is turned "on" prior to actual operation of IAD.

Possible Cause -

1. The microswitch located in the "air inhaled recorder" is actuated. It is mechanically actuated by the recorder-pen-mechanism and may be attributed to dislocation of the bakelite cam, or actually excessive signal from the tidal air flow sensor which in turn is related to excessive flow of air through the mask assembly.

b. Trouble - Some counters do not respond.

Possible cause -

1. Counter disconnected electrically.
2. The integrator sending the pulsating signal is malfunctioning or may be damaged. Examine with a simple volt meter for pulsating signal from the integrator.

c. Trouble - Detector response is different on "inhale" and "exhale" side.

Possible cause -

1. The sampling capillary tubes are not sampling the same concentration of aerosol.
2. Follow Steps 4 through 10 of Operating Procedure to bring the signals into balance.

d. Trouble - The "agent retained" recorder does not indicate the true difference in signal magnitude as is shown on "inhalation" and "exhalation" recorders.

Possible cause - The signals have been thrown off balance by turning of the signal coordination control knob which is located just behind the mask assembly. Make the necessary adjustment.

e. Trouble - Air inhaled recorder does not respond.

Possible cause -

1. Sensor disconnected or damaged.
2. Zero adjust knob (front panel) was turned too far to the left.
3. The mass flowmeter sensitivity adjustment screw, located inside of the recorder was turned to the left-adjust where necessary.

AD ____________ Accession No. ______

General American Transportation Corp.,
MRD Division, Niles, Ill.

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by M. W. Norell, J. D. Zeff
Final Report
22 Dec., 1961 to 15 May 1962
pp. illus. tables
Contract No. DA-18-108-CML-7006

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