R.P.I. Technical Report MP-19

CHROMATOGRAPHIC TEST FACILITY

Steven R. Baer and Gordon L. Benoit

National Aeronautics and Space Administration

Grant NGL 33-018-091

Analysis and Design of a Capsule Landing System and Surface Vehicle Control System for Mars Exploration

MARCH 1971

Rensselaer Polytechnic Institute
Troy, New York

ABSTRACT

The initial unmanned missions to Mars will require a gas chromatograph/mass spectrometer system to make determinations concerning the existence of certain chemical and biochemical species on the planet. It is the overall objective of this task to generate fundamental engineering design techniques and concepts for use in optimizing the design of the chromatograph. This particular subtask has as its objective the building of a test facility for use in verifying the mathematical models of the chromatograph, for obtaining chromatographic data, and for testing possible system concepts.

A previously used commercial chromatographic system was rejuvenated and modified. New features include a pair of micro-thermal conductivity detectors for monitoring inlet and outlet gas compositions, an improved system for reproducibly injecting gas and liquid samples, and an oscillograph for recording data. To obtain accurate recordings of the transient behavior of the gas composition, the system dynamics were improved by reducing detector time constants and dead volume within the piping. The system has been completed and is currently being used to obtain chromatographic data.

CONTENTS

LIST OF FIGURES

(Presented in sequence at the end of the report)

LIST OF TABLES

ACKNOWLEDGEMENT

The authors express their sincere thanks to Professor P.K. Lashmet for his help through various portions of the project. His advice and suggestions proved to be of invaluable assistance in completing the objectives set forth. We also thank the many classmates who helped to accomplish portions of the project. Special thanks are expressed to Kenneth Blanchard and to Joseph Papa who lent assistance in the design and building of electrical circuits associated with the project. The interest and suggestions of Mr. J. Moore, of JPL, the contract monitor, are sincerely appreciated.

I. INTRODUCTION

One of the principle objectives of the unmanned Mars roving vehicle is to determine whether life has, can, or does exist on the planet. It is known that for life to exist, a pre-requisite condition is the presence of certain chemical species. In searching for such clues of life, samples of the atmosphere and soil will be subjected to certain chemical and biological treatments. Gases and liquids resulting from these experiments will be analyzed for the basic chemical species[1,2,3].

It has been shown that a combination GC-MS is applicable for this chemical analysis[4,5]. It is the function of the gas chromatograph to perform an initial separation of a sample into groups, consisting of different species with similar chemical properties. The final separation and molecular weight determinations are made by the mass spectrometer. In addition to its separating function, the gas chromatograph is capable of provided the relative concentrations and the gross physical characteristics of each species present in the sample, although positive identification is not always possible.

Currently a task is being undertaken to generate fundamental engineering design techniques and concepts for use in optimizing the design of the chromatographic separation system. Because of the complexity of the system and the number of independent parameters, a systems analysis based on the mathematical simulation of the chromatograph is being conducted. This technique uses mathematical models, which incorporate the fundamental parameters, to explore various concepts and to direct experimental research. Prior studies[1,6,7] have developed mathematical models based on the physical processes believed to occur within the chromatograph.

Concurrently with the theoretical studies, a chromatographic test facility was developed to experimentally evaluate the mathematical models and to define their limitations. The physical characteristics of the chromatograph are the subject of this report.

The facility is based on a Perkin-Elmer Vapor Fractometer, Model 154C, which was used previously in chemical kinetics studies. The fractometer was completely renovated and modified to provide reproducible data under controlled conditions. The system is operational and chromatographic data are being obtained to verify the mathematical models.

II. GENERAL SYSTEM DESCRIPTION

It is the purpose of the gas chromatograph to separate a gaseous mixture into various subgroups by adsorption and desorption. In the presence of an inert carrier gas, the various subgroups have different dynamic sorption characteristics for a solid or liquid adsorbent. These different characteristics are responsible for separation between the species, and the resulting changes in gas compositions can be related to concentrations in the initial mixture[1].

The gas chromatograph consists of six basic elements:

1. A source of carrier gas and a means for regulating it.
2. A sample injection system and adsorption columns for separation.
3. A means for detecting composition changes.
4. An amplifier for the detector signal.
5. A signal recording device.
6. A means for thermally controlling the system.

These elements which were originally present in the Perkin-Elmer system were redesigned to meet the following objectives:

1. The detector response was to be rapid so signals accurately represented the time-composition behavior of the carrier gas.
2. Detection of composition signals at both the inlet and outlet of the chromatographic column was required.
3. The signal recorder was not to limit the time response of the system.
4. A minimum of sample dead time, thermal transients, and detector drift was desired to provide reproducible and accurate data necessary for verifying the mathematical models.

The system is not to simulate a chromatograph suitable for the Mars mission[8], but is to provide model verification data and a means for examining concepts developed from the theoretical studies.

The completed system is shown in Figure 1. As seen in the system flow diagram, Figure 2, helium is used as the carrier gas. Helium, as well as being inert toward various adsorbents suitable for the chromatographic columns, has a high thermal conductivity, which makes it a useful reference for the thermal conductivity cells which are used as composition detectors. The helium which is generally of high purity (99.95% pure) to insure against extraneous detector signals, is regulated and metered. Pressures range from 2 to 5 atm abs, and flow rates range to about 200 ml (STP)/min.

The carrier gas enters the oven chamber and its temperature is raised to the operating temperature of the system by the preheater. The gas now passes through the reference side of both the input and output composition detectors. This effectively eliminates all errors arising from impurities in the carrier gas, and also possible errors from a small temperature difference between the detector chamber and the carrier gas.

The carrier gas is then split into two streams, one going to a mechanical sample injection system (Carle Mini-Valve), and the other stream passing through a conventional injection block suitable for syringe injection. In the injection valve system, a sample gas to be injected is first preheated in the oven chamber, then regulated before entering the sample loop which has a volume of 100 μl. When the valve is switched, the sample is injected into the carrier gas stream. The other portion of the carrier gas stream passes through a standard

injection block which contains a small heating coil for vaporizing liquid samples. A syringe containing the sample (either gas or liquid), is inserted through a silicone rubber septum into the block, where the sample is injected into the carrier gas stream. The two streams are combined, and the carrier gas containing the sample enters the input detector.

After leaving the inlet detector, the sample containing carrier gas enters the chromatographic columns. Although initial experiments will involve single columns, the column mounting, as seen in Figure 4, is designed to accomodate two columns, arranged either in series or in parallel. The columns may be 3.2 or 6.4 mm in diameter and up to one meter in length. The gas mixture, after being separated, passes through the outlet composition detector and is then vented or scrubbed if hazardous materials are used.

The detectors, columns and injection devices are enclosed within a forced air convective oven. The oven temperature is controlled to about 1 deg C within its operating range of 50 to 225 deg C.

Considerable modification of the original Perkin-Elmer chromatograph was required to produce the required testing facility described above. Major changes and specific details about each of the major areas in the system are documented in the following sections.

III. DETECTORS AND CONTROL

To obtain the required response characteristics over a wide range of sample mixtures, a thermal conductivity detector was chosen for this system[9,10]. These detectors are simple and reliable, and can be used in the presence of many different kinds of sample gases. As seen schematically in Figure 2, each detector has two similar chambers. Through one chamber flows the carrier gas before sample injection, and through the other flows the carrier gas plus the sample being analyzed. In each chamber is a thermistor through which a regulated current flows. The electrical resistance of the thermistor is a direct function of its temperature. The gases flowing through the thermistor chamber conduct heat away from it and thereby change the resistance. The rate at which the gas conducts heat away from the thermistor depends on the thermal conductivity of the specific gas mixture flowing through the chamber. Each gas has a unique thermal conductivity which is approximately inversely proportional to its molecular weight.

Since the sensing chamber has flowing through it gases of molecular weights different from that of the pure carrier gas, the overall thermal conductivity of the gas in the sensing chamber will be different from the conductivity of the reference chamber, which contains only pure carrier gas. Helium was selected as the standard or carrier gas because its thermal conductivity is far different from most gases to be analyzed as seen in Table I[11], and it is available in high purity. Because only hydrogen has a higher thermal conductivity than helium, in most experiments of interest the thermal conductivity in the sensing chamber will be lower than the reference conductivity so the detector output

TABLE I

THERMAL CONDUCTIVITIES OF TYPICAL GASES

Conditions: 100 deg C and 1 atmosphere pressure

Gas	Thermal Conductivity, watts/(cm, deg C)
Helium	17.1×10^{-4}
Acetone	1.7
Ammonia	3.3
Argon	2.1
Carbon dioxide	2.2
Carbon monoxide	3.0
Hydrogen	21.4
Methane	4.4
Nitrogen	3.1
Oxygen	3.2
Pentane	2.2
Water vapor	2.4

Reference: McAdams, W.H., "Heat Transmission," 3rd ed., pp. 457-458, McGraw-Hill, New York, 1954.

signal will be of one polarity, thereby simplifying signal detection. The thermistor resistances each form legs of a DC Wheatstone bridge circuit, and the difference in voltage drop across each leg generates the output signal which is recorded. The magnitude of the output signal is directly proportional to the amount of sample present in the detecting chamber, at least in the dilute gas mixtures to be considered in this experimental program.

The accuracy and sensitivity of these detectors is directly related to the thermistor bead size, the thermistor chamber volume and its design, and the carrier gas flow rates. After reviewing the sensitivity, dead time, and other characteristics of several commercial detectors, a microdetector manufactured by Carle Instruments, Inc. (Fullerton, California) was selected primarily because of its rapid response and small dead volume. The detector characteristics are summarized in Table II. The Carle detector, which has a time constant in the order of 0.04 second, is an order of magnitude faster than other conventional thermal conductivity detectors. Fast response is desirable in this test facility because the detector signals are to be used in verifying theoretical system models.

As seen in Figure 4, the detectors are physically mounted on an aluminum heat sink at the bottom of the convective oven, and the cell surfaces are thermally insulated from the circulating oven gas by an insulating jacket. This eliminates short-term temperature fluctuations which may cause irregular baseline drift of the detectors.

Because the microdetectors are considerably more sensitive than the detectors originally in the chromatograph and have different operating

TABLE II

DETECTOR CHARACTERISTICS

Manufacturer: Carle Instruments, Inc., Catalog No. 1150

Sensing element:

Bead diameter:	0.005 inch
Cold resistance:	8 - 10 K ohms
Operating resistance:	200 - 500 ohms
Operating current:	20 ma
Wattage dissipation:	0.05

Temperature range: 0 - 250°C

Volume: 58 microliters

Carrier gas flow range: 0.5 - 100 ml/min.

Time constant at 25 ml/min. in He: 0.04 sec.

Connecting tubing: 1/16 inch O.D. x 0.023 in I.D.

characteristics, a new detector control circuit was necessary. This control is basically a Wheatstone bridge having high resolution and a broad control range, and which is powered from a regulated DC power supply. An external view of the controls is given in Figure 3 and the overall circuit diagram is shown in Figure 5. This dual unit is designed to control two detectors at the same time and to produce two recordable output signals. Initially it was thought that both detectors might be controlled using one commercial unit manufacturer by Carle. However, this proved impractical because of switching problems, so a dual control based on the Carle design was constructed.

Both sections of the control unit are identical to one another as seen in Figure 5. Several means for adjustment are provided. An output attenuator divided the detector control output into ten increments over a range of 1000 to 1 so both large and small signals may be recorded. A compensation control adjusts the resistances in the thermistor legs of the circuit so the bridge can be balanced at the highest attenuation ratio for the output signal. Coarse and fine zero controls are ten-turn helical potentiometers with different shunt resistors which zero the output signal at other attenuation ratios. The current adjustment varies the bridge currents to obtain good detector response and a meter is supplied to monitor its value. The multifunctional polarity switch controls power to the bridge as well as selecting the polarity of the DC signal. The test position shorts the recorder input for adjusting the electrical and mechanical zeros of the recorder.

The two parallel control circuits are driven by a single, solid state DC power supply which replaces batteries used in the original unit. A highly regulated 10 VDC voltage is required for proper detector

performance. Characteristics of the selected power supply manufactured by ACDC Electronics, Inc. (Burbank, California) are given in Table III. A meter for monitoring the voltage is provided.

TABLE III

POWER SUPPLY CHARACTERISTICS

Manufacturer:	ACDC Electronics, Inc., Catalog No. BX10NO.3
Input:	105-125 volts, 47-420 Hz
Output:	9 - 11 volts
Current:	0.3 amp
Regulation:	0.01% or 0.001 volt for line change of 10% 0.01% or 0.002 volt for NL to FL
Ripple:	0.5mV or 0.001% max RMS
Stability:	max. 0.1% or 10mV for 8 hour period
Output impedance:	0.001 ohm max.
Temperature coefficient:	max. 0.015% or 1 mV/deg. C.

IV. SAMPLE INJECTION

A system for injecting liquid and gas samples and which provides reproducible results was desirable. The original Perkin-Elmer injection system consisted of an injection block suitable for syringe injection, and a sample preheater for liquid samples. Since liquid syringe volumes are relatively accurate, and liquid injection curves are almost identical, it was decided to leave the sample injection block in the system for liquid sample injection, and to purchase a commercial gas sample injection system.

For liquid samples, a Hamilton liquid syringe with a volume range of 0-10 μl is used. The syringe injects the sample through a silicone rubber septum. The liquid is immediately vaporized in the sample chamber by the sample heater and is swept into the column by the carrier gas.

For gas samples, a mechanical injection value manufactured by Carle Instruments, Inc. (Fullerton, California) is used[12]. Valve specifications are given in Table IV and a schematic of its operation is shown in Figures 6 and 7. As shown in Figure 6, sample gas from a lecture bottle or other sources is preheated and flows through the sample loop of the valve. The gas flow rate is manually controlled by a small needle valve, and the rate is detected by bubbling the gas through water. After passing through the bubbler the sample gas is vented or detoxified.

The amount of sample injected into the system is determined by the size of the sample loop and the gas pressure. Currently the loop volume is 100 μl, but it is adjustable. As shown in Figure 7, the carrier gas flows through the other loop in the valve. When the gas

TABLE IV

INJECTION VALVE CHARACTERISTICS

Manufacturer:	Carle Instruments, Inc., Catalog No. 5518
Configuration:	two position, two stops at 60°, with demountable loop
Loop volume:	100 µl and interchangeable
Temperature range:	ambient to 250 deg C
Pressure range:	ambient to 7 atm abs
Connecting tubing:	1/16 inch O.D. x 0.043 inch I.D.

sample is to be injected, the valve is turned, changing the polarity of the valve. This causes the carrier gas to be diverted through the sample loop. The sample gas in the loop is swept into the column by the carrier gas.

The two systems are in parallel to eliminate additional piping. A switching valve was considered but was rejected because of additional joints for possible leakage and general unreliability.

To preserve as well as possible the dynamic characteristics of the carrier gas compositions external to the chromatographic columns, the holdup times in the piping of the injector and detector systems are kept as small as possible. Figure 8 summarizes delay times in the various lines for a typical carrier gas flow of 40 ml (actual) per minute. It is noted that the delay times are small compared with the transport time in a one meter chromatographic column which generally is used in this experimental program. Detector signals therefore represent fairly well the actual time behavior of the gas compositions entering and leaving the column.

V. OVEN AND THERMAL CONTROL

Gas chromatography is generally conducted at either a constant temperature, or at temperatures which vary in a specified manner[13]. For this reason the column, detector, and injection valve are housed in a constant temperature oven. The oven is an asbestos lined, aluminum chamber with internal aluminum baffles. The oven is heated by a coil heating element, and a fan is used to circulate the heated air throughout the oven chamber. Gas temperatures are monitored by a gas bulb thermometer located centrally in the oven, and three iron-constantan thermocouples, positioned in the chamber indicate the vertical temperature profile. The oven temperature is controlled by a thermostatic amplifier using a thermistor in the oven as a sensing unit. The amplifier shown schematically in Figure 9 is a two stage, on-off controller which manipulates an externally adjustable load at the heating coil as seen in Figure 10. The temperature range is ambient to 225°C. Coarse and fine controls adjust the temperature set point and control sensitivity by changing the control circuit resistances. Maximum power to the heater is manually adjusted by a power transformer. This can be used to adjust for gross changes in the ambient temperature and for initially heating the oven to operating conditions. With this system, oven temperatures are controlled to within about 1 deg C, which is adequate for the chromatographic data to be obtained.

Except for rejuvenation, the present oven controller is the same as the original unit. Major control relays and several electrolytic capacitors were replaced, and incorrect wiring was eliminated.

VI. CARRIER GAS FLOW REGULATION AND MEASUREMENT

An important parameter of column operation is the flow rate of the carrier gas through the column. Therefore regulation of the carrier gas flow rate is necessary. Referring to the flow diagram, Figure 2, it is seen that a pressure regulation valve is in the line with the incoming gas. The pressure is regulated in the range of 2 to 5 atm abs, and is monitored by a pressure gauge mounted after the pressure regulator. Flow rate to a maximum of 200 ml (STP)/min is monitored with a conventional rotameter manufactured by Brooks Instruments, Inc., (Hatfield, Pennsylvania). The rotameter (Type 1555-00A1AAD) uses a 15 cm graduated glass tube (Type R-2-15-AAA) and a stainless steel ball float. Calibration curves for the rotameter using helium are shown in Figure 11.

The original carrier gas piping has been completely replaced. Stainless steel tubing (0.250 inch O.D. by 0.152 inch I.D.) and ferrule-type fittings (Gyrolock fittings) have been used throughout to reduce possible corrosion problems. Whenever practical, the tubing was bent to avoid extra fittings and potential leakage. The vent to the atmosphere is valved to pressurize the system with helium when it is not in use to avoid condensation of water in the lines, and to keep the detector chambers calibrated.

VII. RECORDER

The composition detectors of the test facility produce an output voltage proportional to the composition of a given species present in the detector chamber as a function of time. Because these signals are to be used in verifying mathematical models, it is necessary to obtain a signal recording which accurately represents the time response of the detectors. The originally available recorder, a self balancing potentiometer (Leeds and Northrup Speedomax Recorder, Type G), while being adequate for general analytical use, was too slow in responding with its one second full scale deflection time. In the final system, the input and output signals are recorded with a Honeywell oscillograph having the characteristics given in Table V. Comparison of the galvanometer time response with the time constant of the composition detector shows the detectors are limiting the response of the system, a desirable feature.

TABLE V

HONEYWELL OSCILLOGRAPH - MODEL 906-1 OPERATING CHARACTERISTICS

NUMBER OF CHANNELS	14
RECORDING SPEEDS	.2,1,5,25 inches per second
FREQUENCY RANGE	0-5000 cps
TIMELINES	.01, .1,1 second per line
CROSSTALK	less than 1% between channels
GRIDLINE SYSTEM	calibrated reference lines .1 inches apart
RECORDING PAPER	direct print type, 6" wide
GALVONOMETERS	type no. M100-350 undamped natural frequency - 100 HZ flat frequency response range - DC-60HZ maximum safe current - 10 ma dampening - electromagentic type max. linear deflection - 8 inches external damping resistance - 350 ohms

VIII. PRELIMINARY DATA

The results of preliminary experiments with several different systems are shown in Figures 12 and 13. Figure 12 shows typical injection curves obtained from the different injection techniques available in the equipment. The ordinates are proportional to gas composition, and the data have been numerically normalized so the areas under each curve are the same. This is equivalent to using identical sample sizes (on a molar basis). Manual injection with the syringe causes the curves to be skewed and to have much tailing. Preliminary experiments show that liquid injection is somewhat more reproducible than gas injection. However, in either case, the injection curves are not similar to the impulse or rectangular pulse inputs assumed in the modern theoretical treatments of the chromatographic process[14]. On the other hand, the Carle valve provides a composition signal which simulates the rectangular pulse and which is reproducible.

The output signals or chromatograms for the three inputs are shown in Figure 13. The columns used for the three systems were one meter long by 3.2 mm in diameter. The diameter of the solid particles within the column ranged between 0.18 mm to 0.25 mm (60/80 mesh). Again the ordinates are proportional to gas composition and the data have been normalized. The data for the air/molecular sieve system correspond well with literature values[15,16].

IX. CONCLUSIONS

From the preliminary experiments, it is concluded that the test facility is capable of generating repeatable gas chromatographs which will be suitable for verifying the theoretical studies as well as for investigating various system concepts. The rapid response of the detectors and recorder to composition changes within the system and the small dead times in the system piping give confidence that the recorded data represent far more accurately the actual time response than can be obtained in conventional chromatographs.

X. REFERENCES

1. Sliva, T.F., "Chromatographic Systems Analysis: First-Order Model Evaluation," R.P.I. Technical Report MP-1, Rensselaer Polytechnic Institute, Troy, New York, September, 1968.

2. Moore, J.W., "An Exploratory Investigation of a 1979 Mars Roving Vehicle Mission," Report 760-58, Jet Propulsion Lab, Calif. Inst. Tech., Pasadena, December 1, 1970.

3. Kiely, J.R., ed., "SAMPLER: Stanford Advanced Mars Project for Life Detection, Exploration and Research," School of Engineering, Stanford University, June 1965.

4. Oyama, V.I., "Use of Gas Chromatography for the Detection of Life on Mars," Nature, 200, 1058-1059 (1963).

5. "Bentley, K.E., Griffen, C.E., Whitten, D.G., and Wilhite, W.F., "Detection of Life-Related Compounds on Planetary Surfaces by Gas Chromatography - Mass Spectrometery Techniques," Tech. Report 32-713, Jet Propulsion Lab., Calif. Inst. Tech., Pasadena, August 1965.

6. Voytus, W.A., "Chromatographic Systems Analysis: Moment Analysis of the Equilibrium Adsorption Model," R.P.I. Technical Report MP-9, Rensselaer Polytechnic Institute, Troy, New York, August 1969.

7. Taylor, P.N., "Chromatographic Systems Analysis: Second-Order Model Development," M.Eng. Report, Rensselaer Polytechnic Institute, Troy, New York, June 1970.

8. Wilhite, W.F., "Developments in Micro Gas Chromatography," Tech. Report 32-805, Jet Propulsion Lab., Calif. Inst. Tech., Pasadena, March 1, 1966.

9. Lawson, A.E., Jr., and Miller, J.M., "Thermal Conductivity Detectors in Gas Chromatography", J. Gas Chromatog., 4, 273-284 (1966).

10. Rosie, D.M., and Grob, R.L., "Thermal Conductivity Behavior: Importance in Quantitative Gas Chromatography," Anal. Chem. 29, 1263-1264 (1957).

11. McAdams, W.H., "Heat Transmission", 3rd ed., pp. 457-458 McGraw-Hill, New York, 1954.

12. McKinney, C.B., "Selector Valve", U.S. Patent 3,297,053 (January 30, 1967).

13. Harris, W.E., and Habgood, H.W., "Programmed Temperature Gas Chromatography", Wiley, New York,1966.

14. Yamazaki, H., "A Mathematical Treatment of Nonequilibrium Chromatography", J. Chromatog., 27, 14-19 (1967).

15. Parkinson, R.T., and Wilson, R.E., "Elution of Oxygen from a Highly Activated Molecular Sieve 5A Column," J. Chromatog., 36, 551-553 (1968).

16. Walker, J.A., "Chromatographic Separation of Argon and Oxygen Using Molecular Sieve," Nature, 209, 197 (1966).

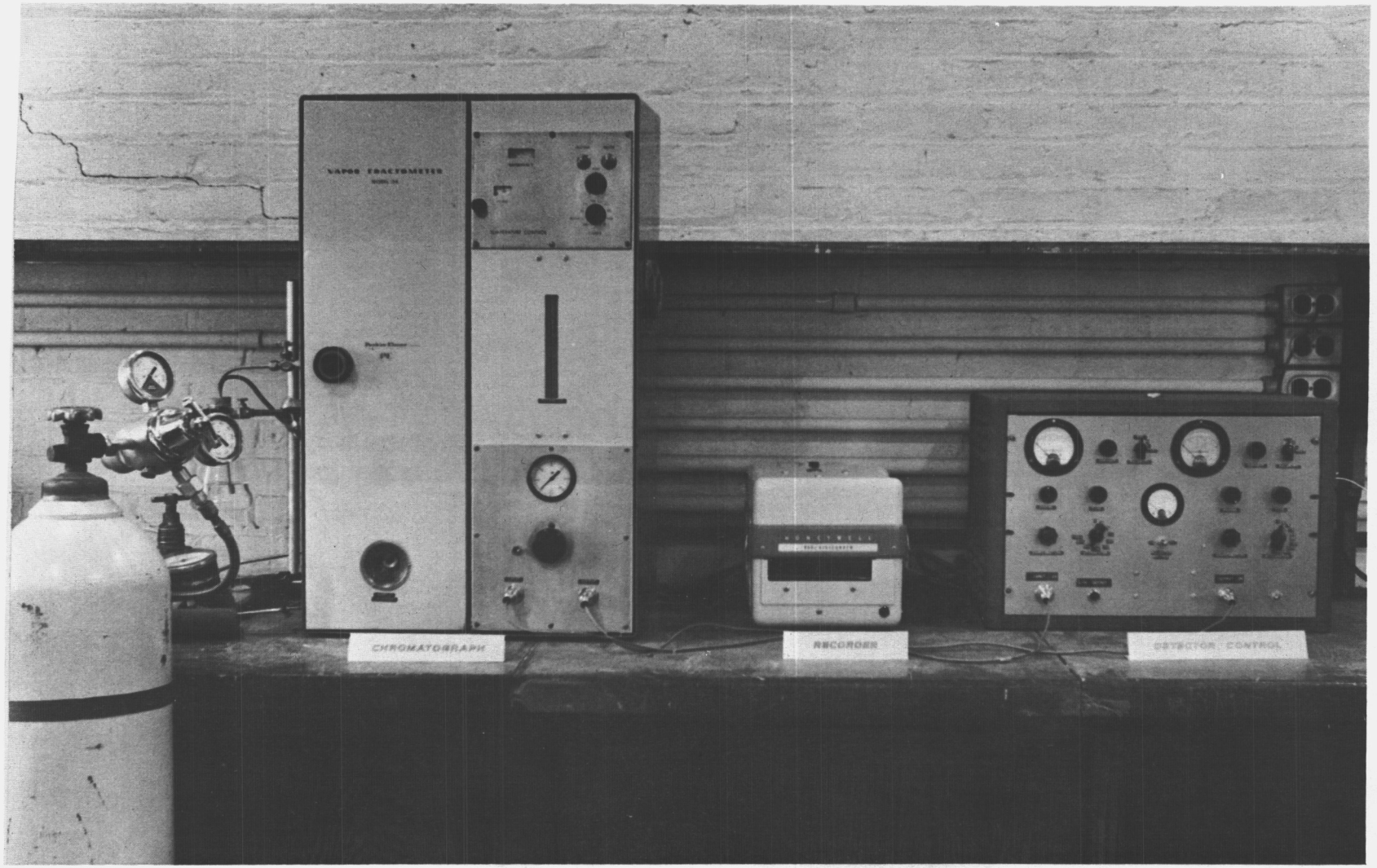


FIGURE I. CHROMATOGRAPHIC TEST FACILITY

SYSTEM FLOW DIAGRAM

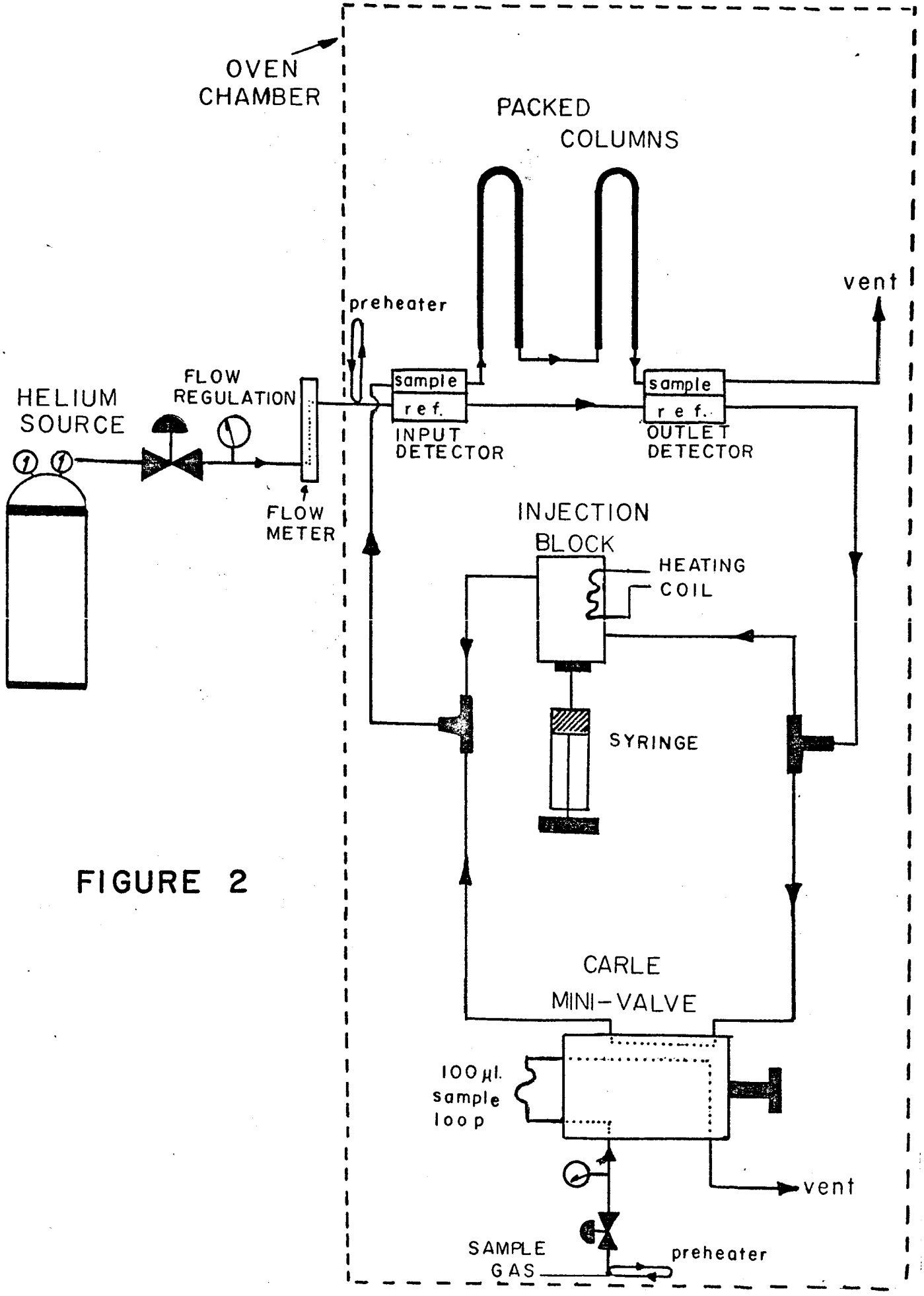


FIGURE 2

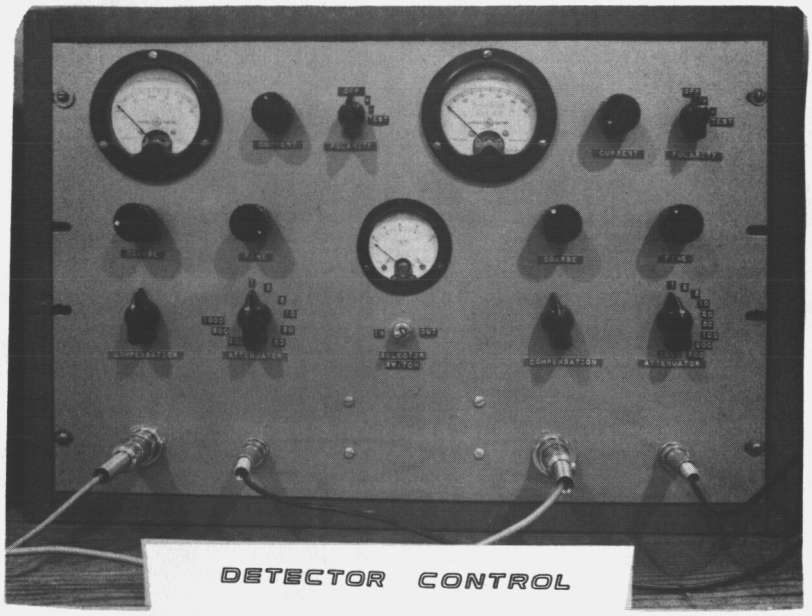


FIGURE 3. DETECTOR CONTROL

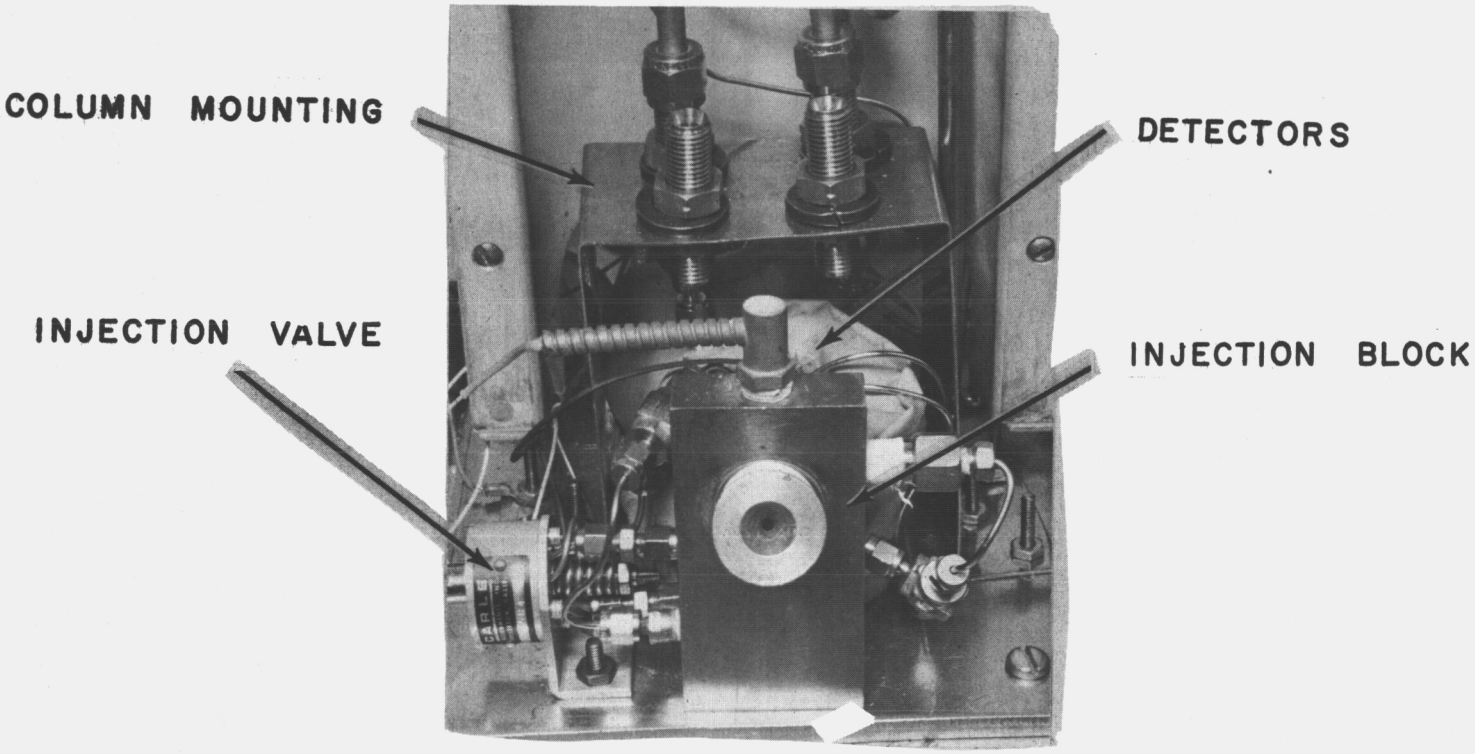
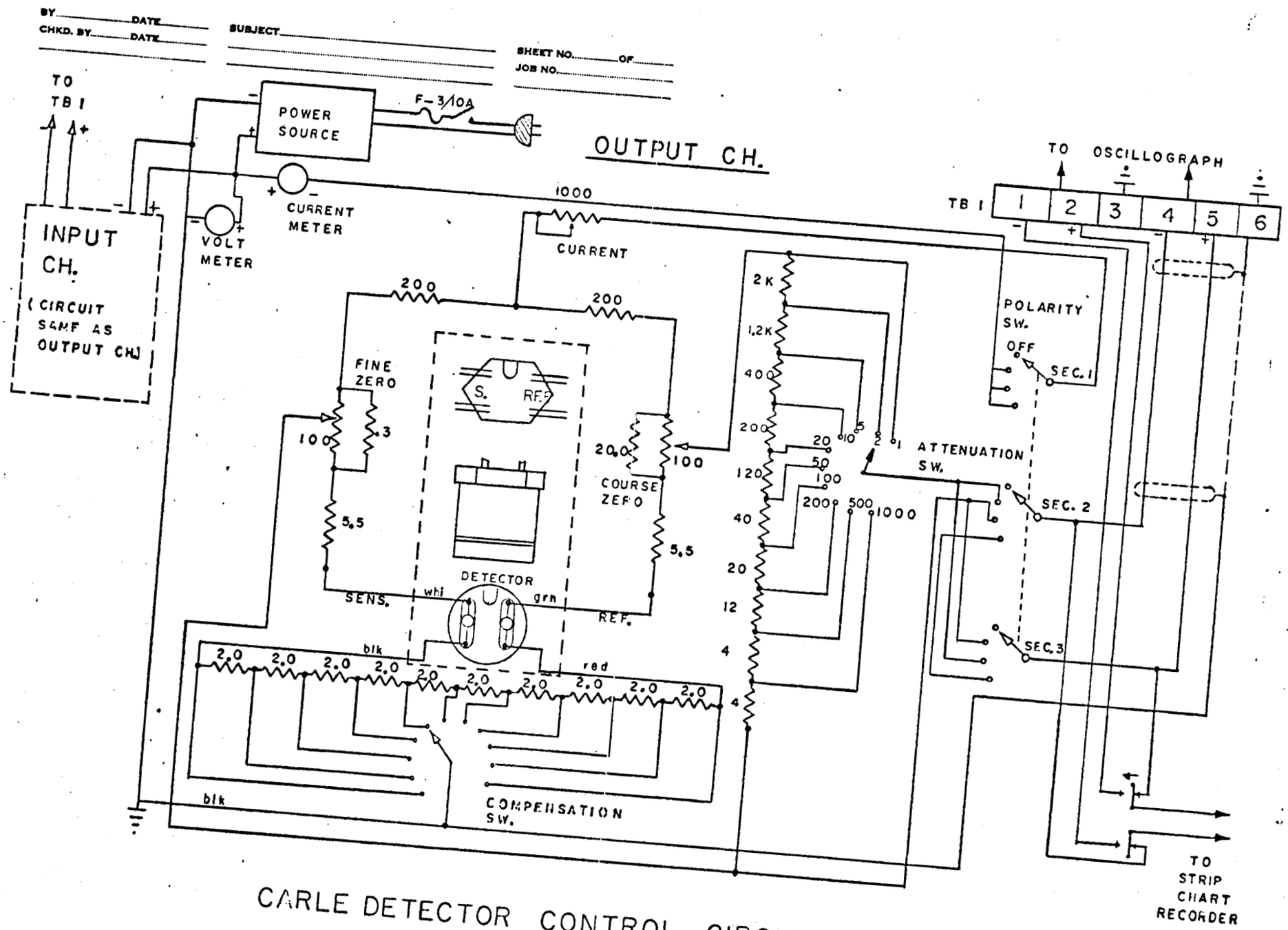


FIGURE 4. DETECTORS AND SAMPLE INJECTION SYSTEM



CARLE DETECTOR CONTROL CIRCUIT — FIG. 5

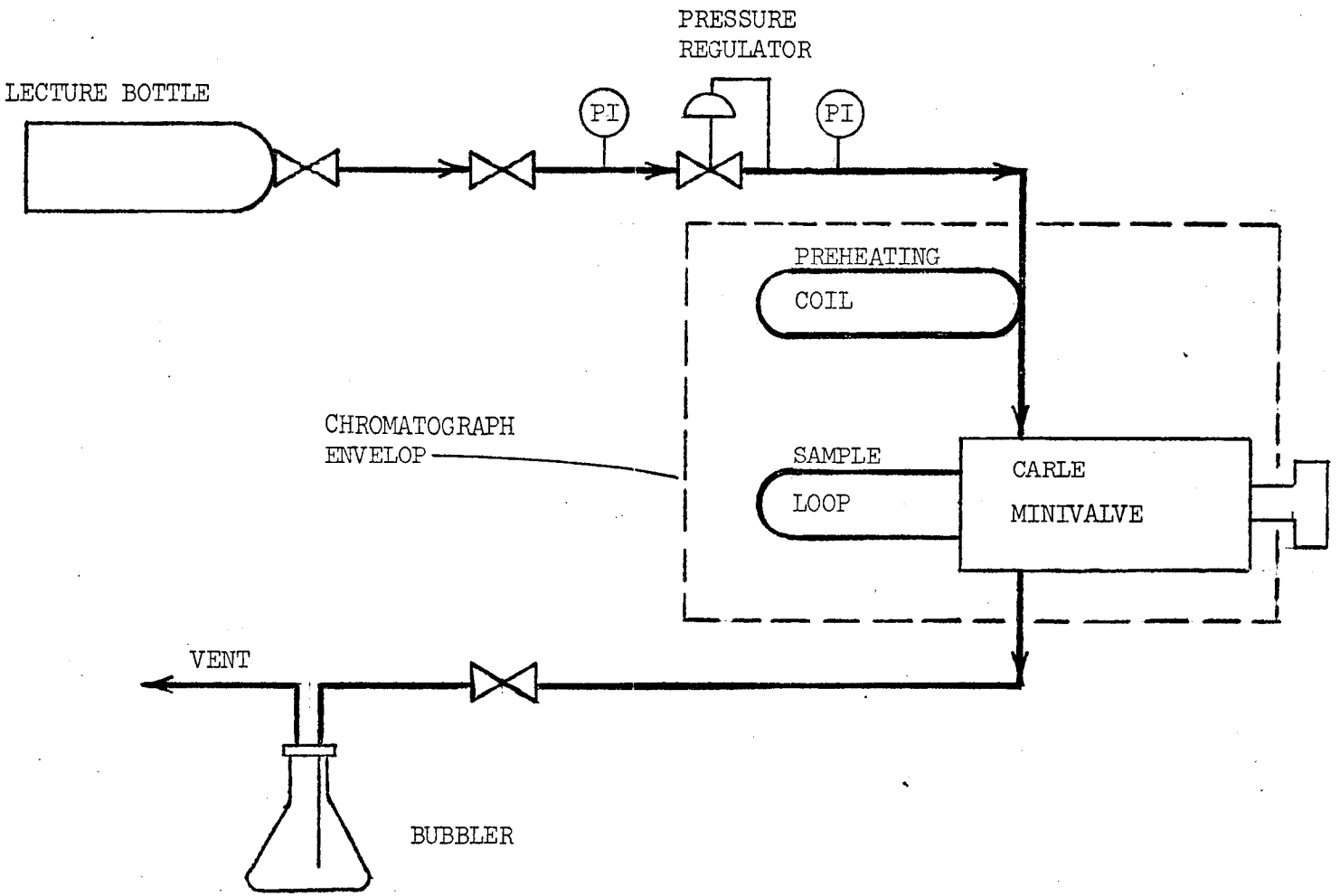
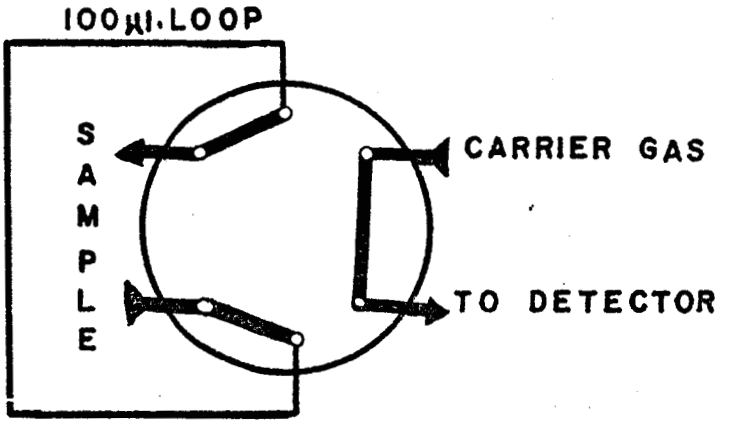


FIGURE 6 SAMPLE INJECTION SYSTEM USING CARLE MINI-VALVE

CARLE MINI-VALVE OPERATION

BEFORE INJECTION



AFTER INJECTION

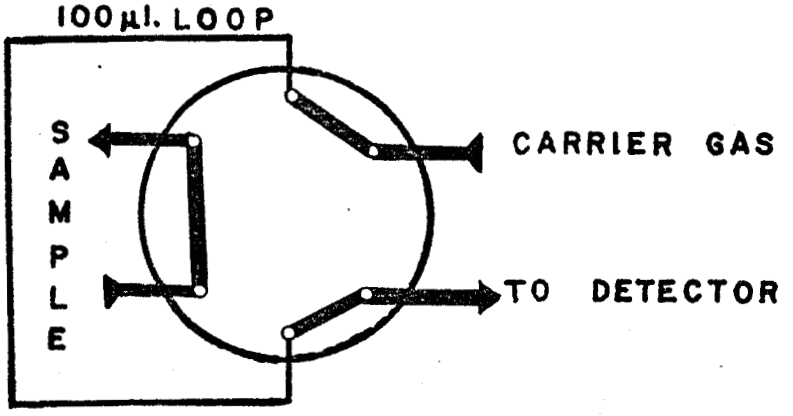


FIGURE 7

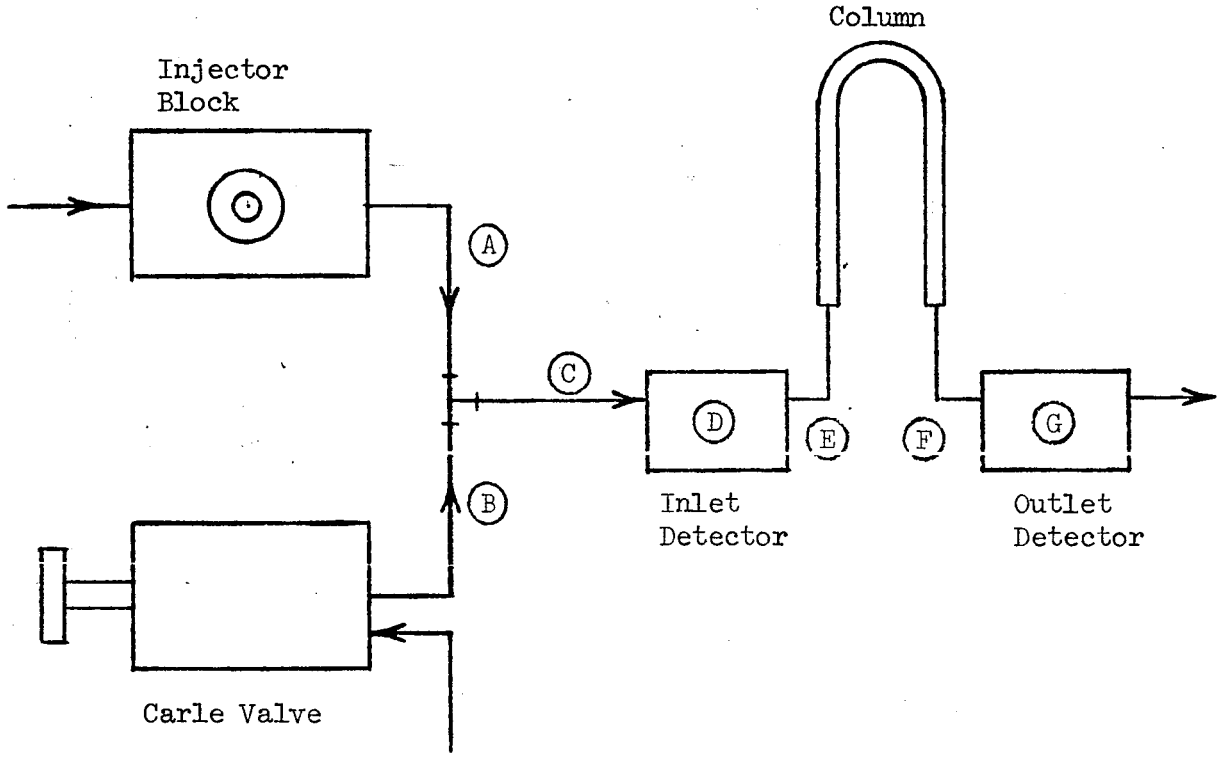


FIGURE 8 ESTIMATED DEAD TIME IN PIPING

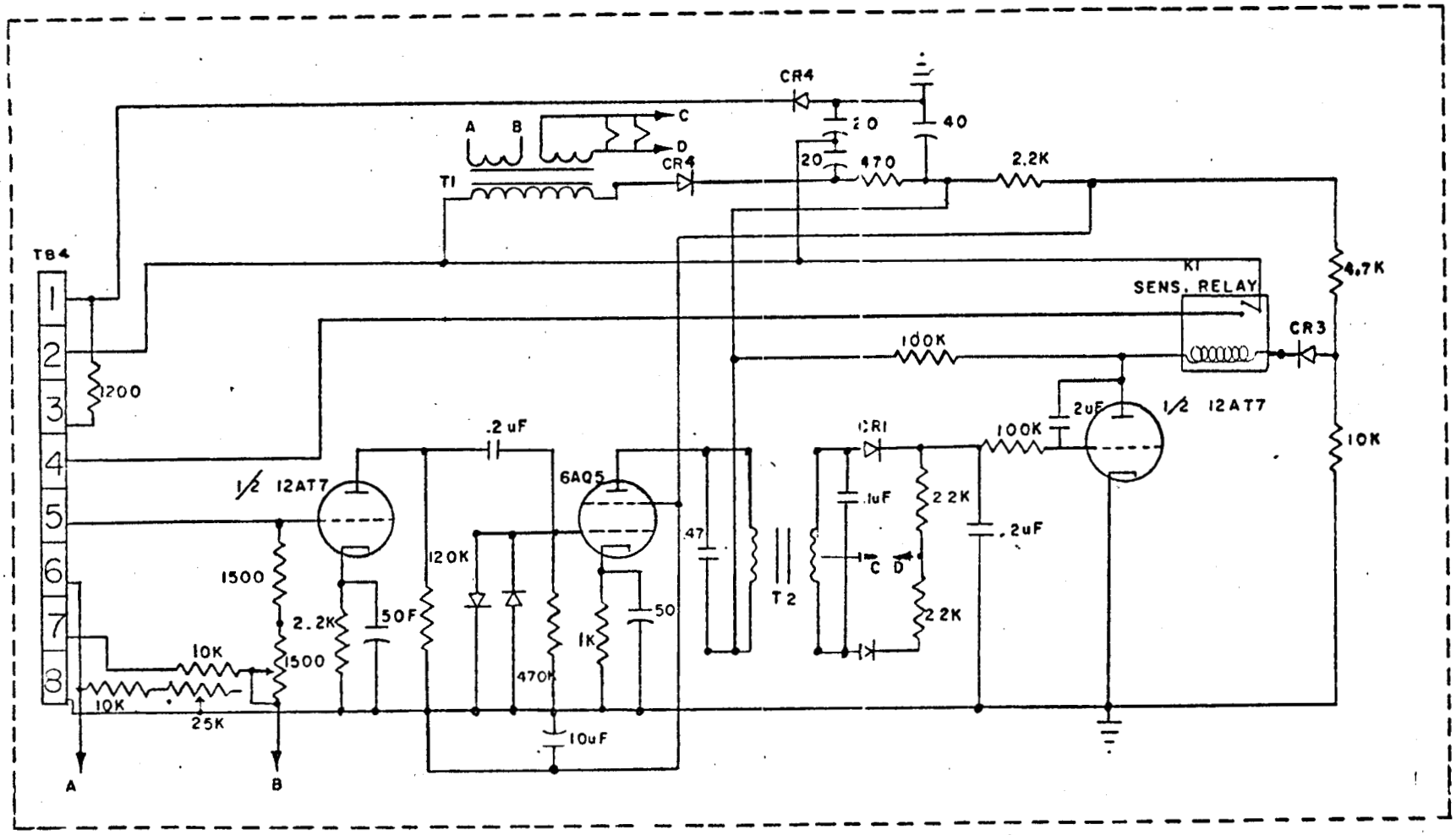
Conditions: helium flow at 40 ml(actual)/min

A	- 0.02 second	E	- 0.02 second
B	- 0.03 second	F	- 0.02 second
C	- 0.01 second	G	- 0.09 second
D	- 0.09 second		

Typical column (1 meter long by 2.2 mm I.D.) - 2.2 seconds

BY_____DATE_____ SUBJECT_____ SHEET NO._____OF_____

CHKD. BY_____DATE_____ JOB NO._____

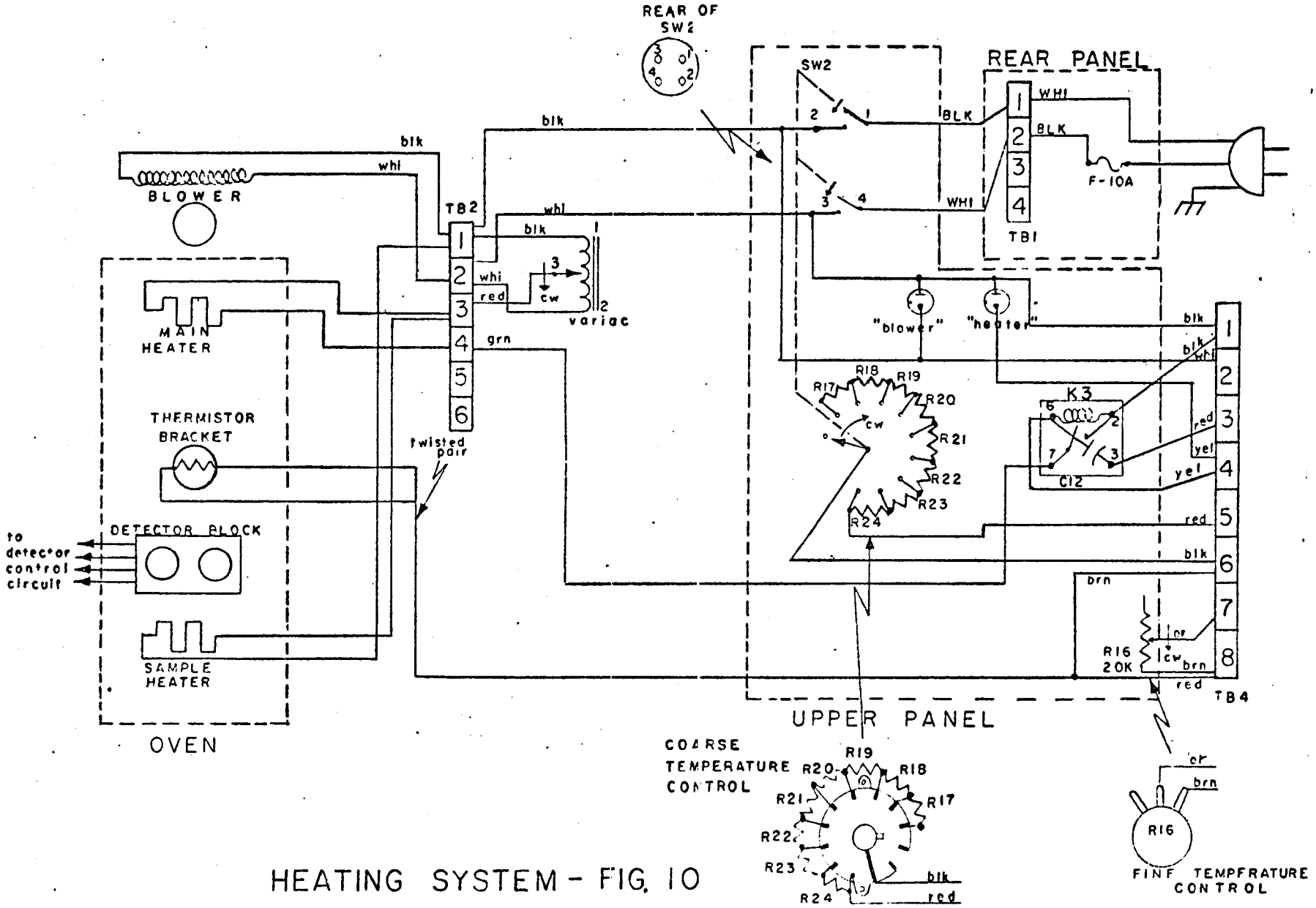


THERMOSTAT AMPLIFIER

HEATING SYSTEM-FIG. 9

BY_____ DATE_____ SUBJECT_____ SHEET NO._____ OF_____

CHKD. BY_____ DATE_____ JOB NO._____



HEATING SYSTEM - FIG. 10

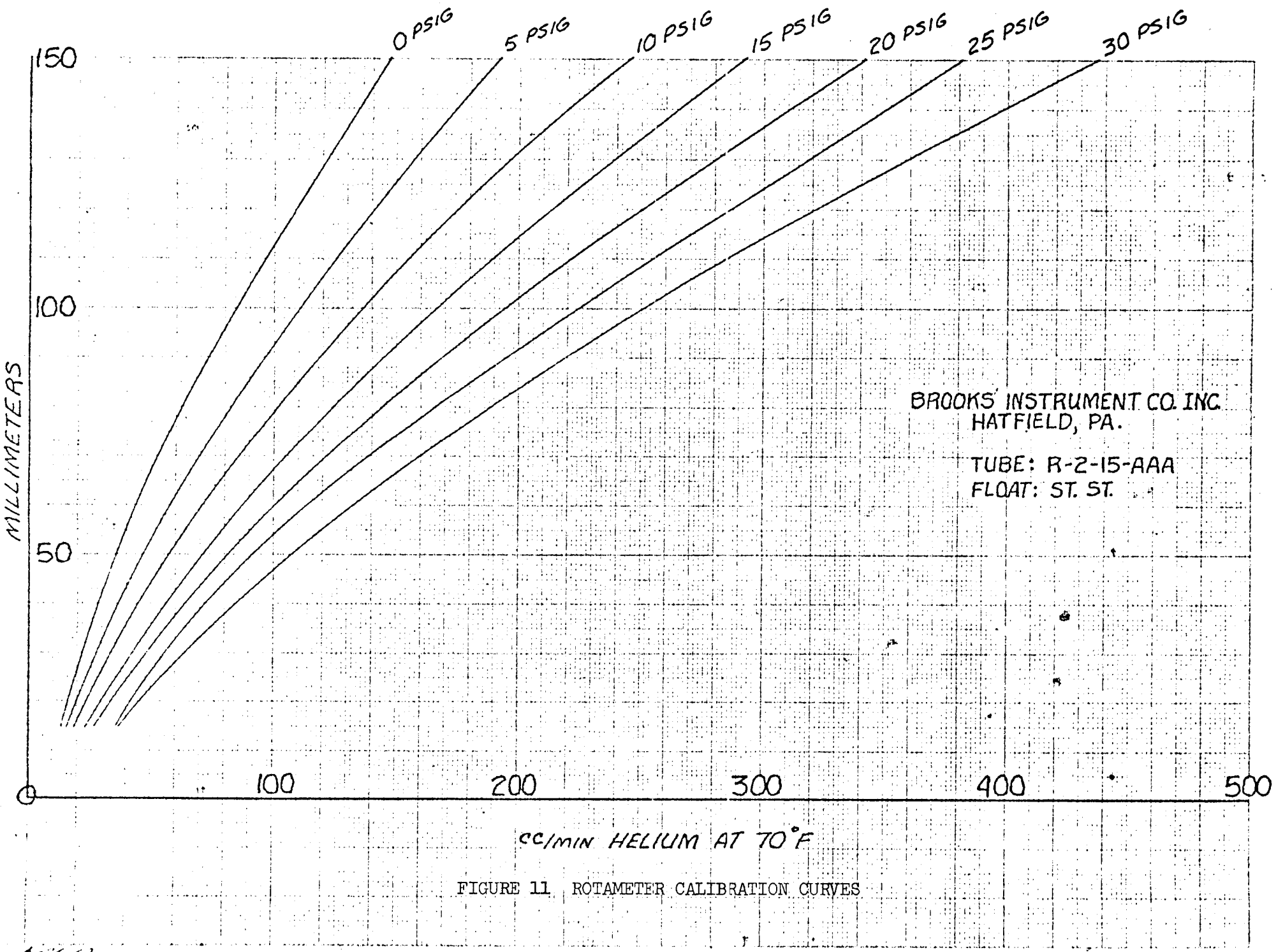
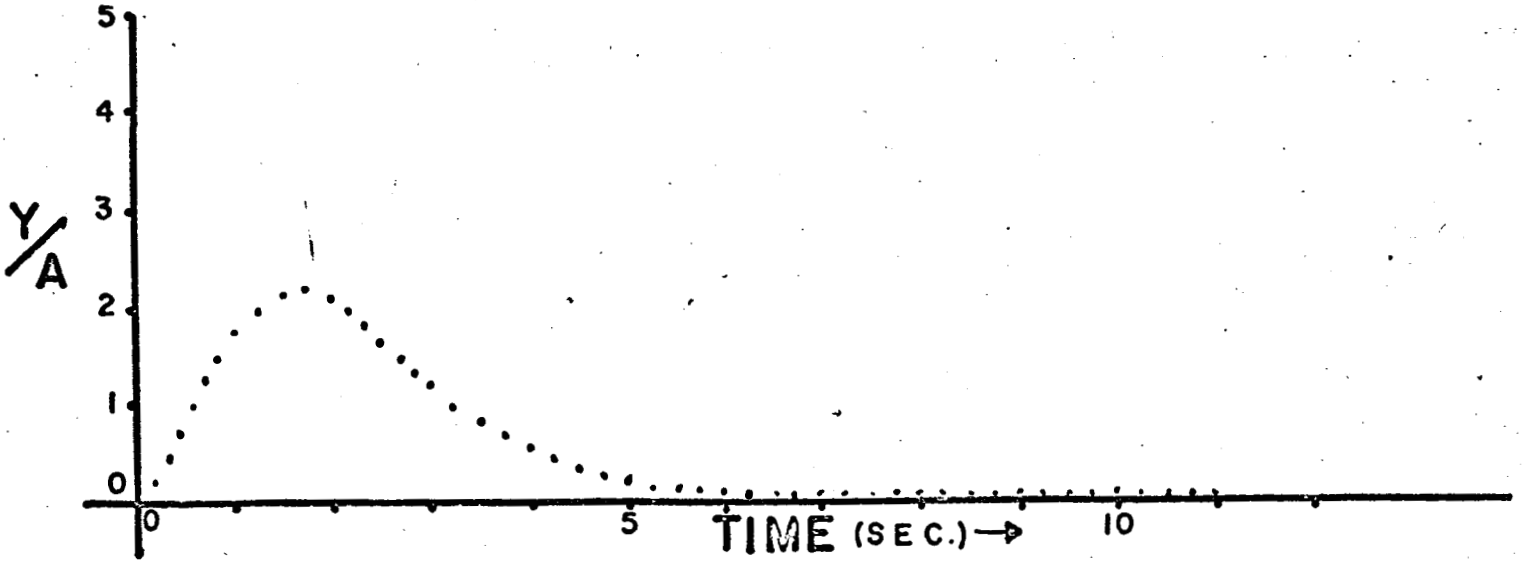
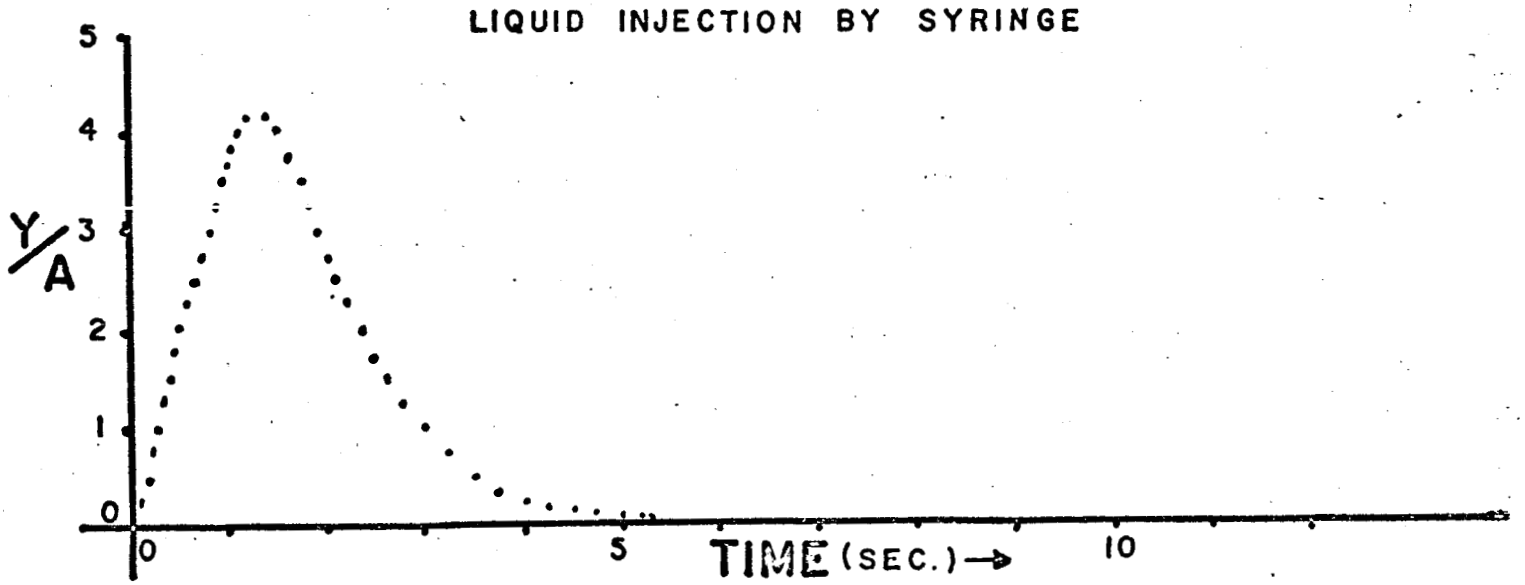


FIGURE 11 ROTAMETER CALIBRATION CURVES

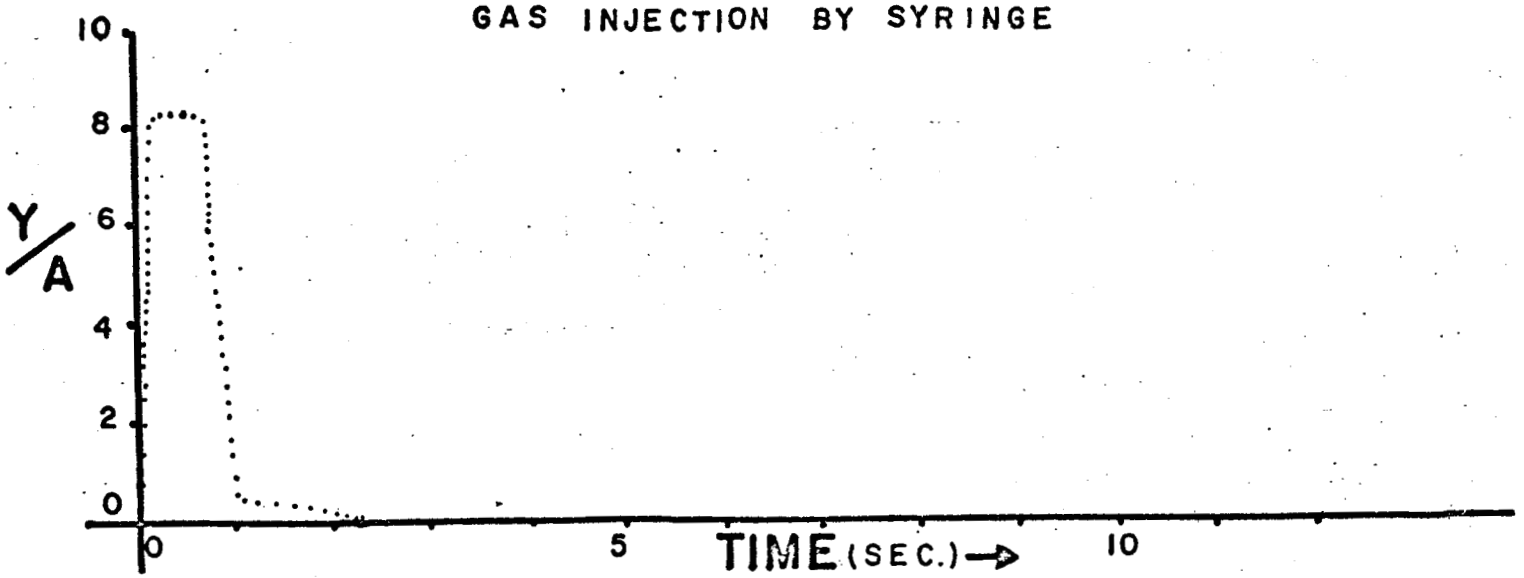
COMPARISON OF INJECTION TECHNIQUES



LIQUID INJECTION BY SYRINGE



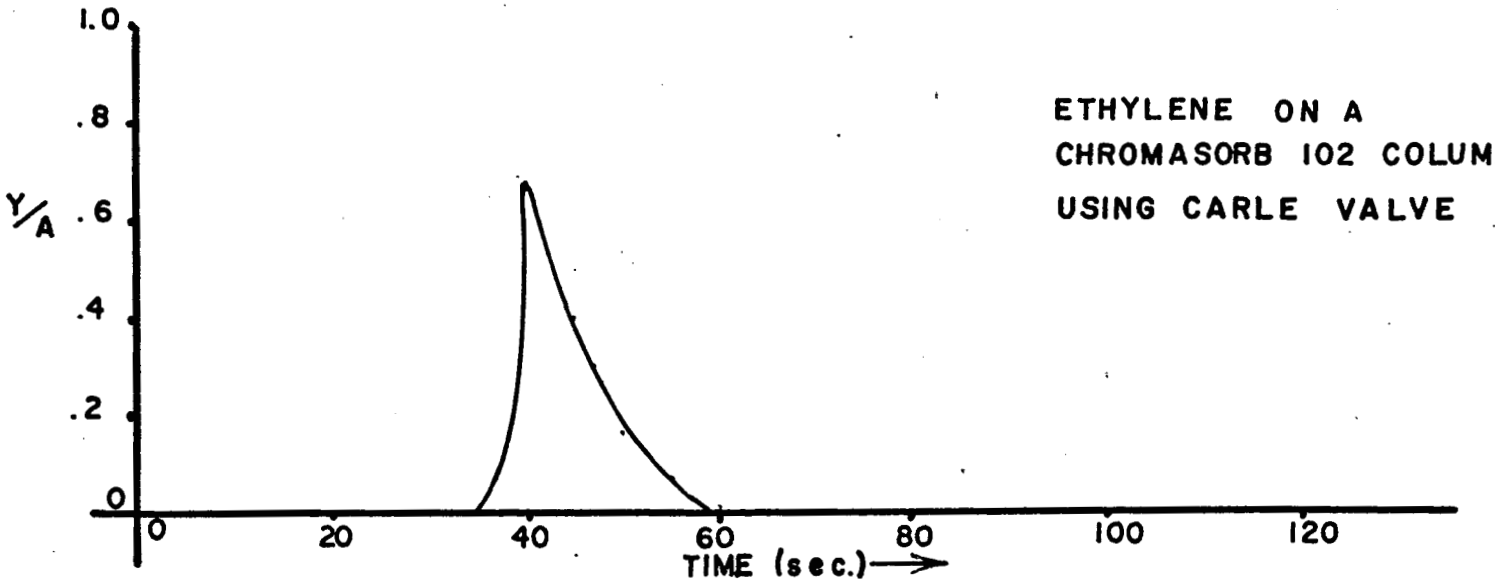
GAS INJECTION BY SYRINGE



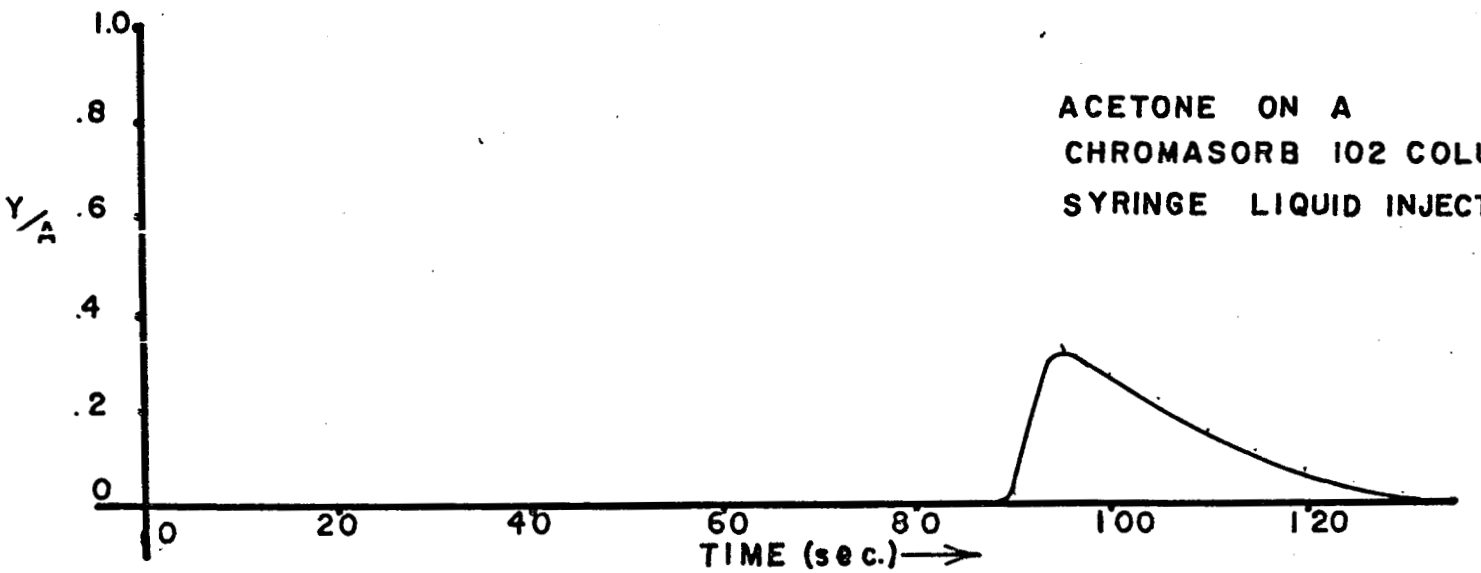
GAS INJECTION BY CARLE MINI-VALVE

FIGURE 12

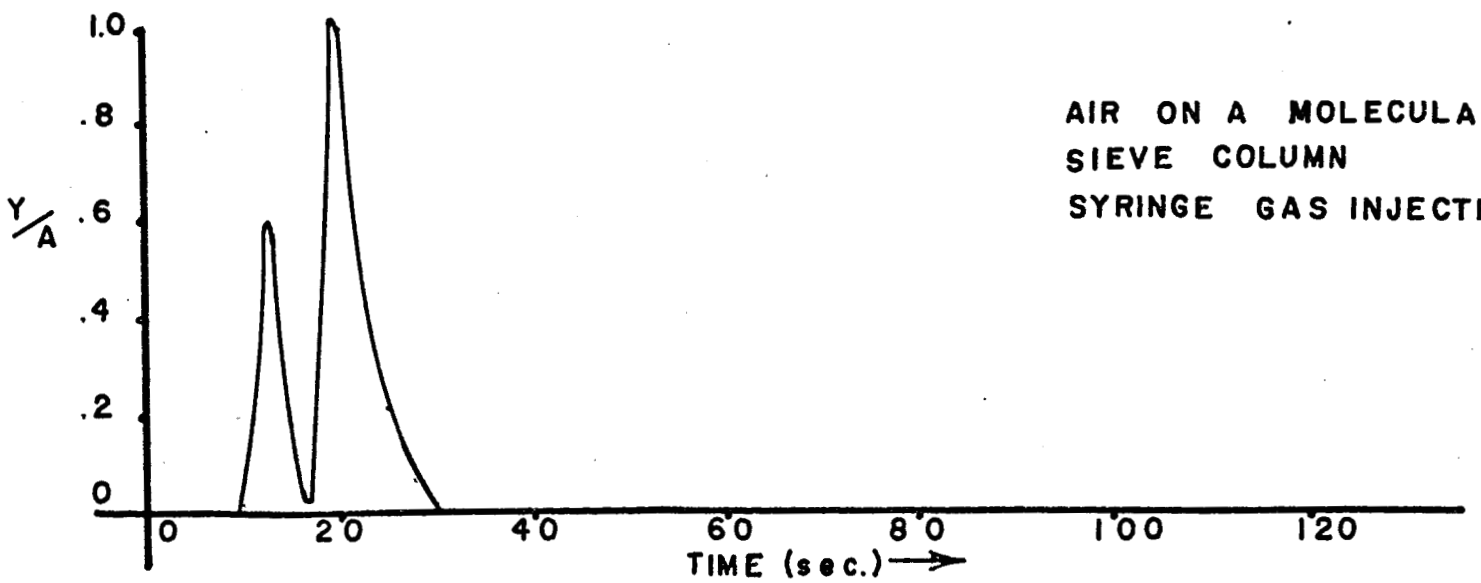
SAMPLE GAS CHROMATOGRAMS



ETHYLENE ON A
CHROMASORB 102 COLUMN
USING CARLE VALVE



ACETONE ON A
CHROMASORB 102 COLUMN
SYRINGE LIQUID INJECTION



AIR ON A MOLECULAR
SIEVE COLUMN
SYRINGE GAS INJECTION

FIGURE 13