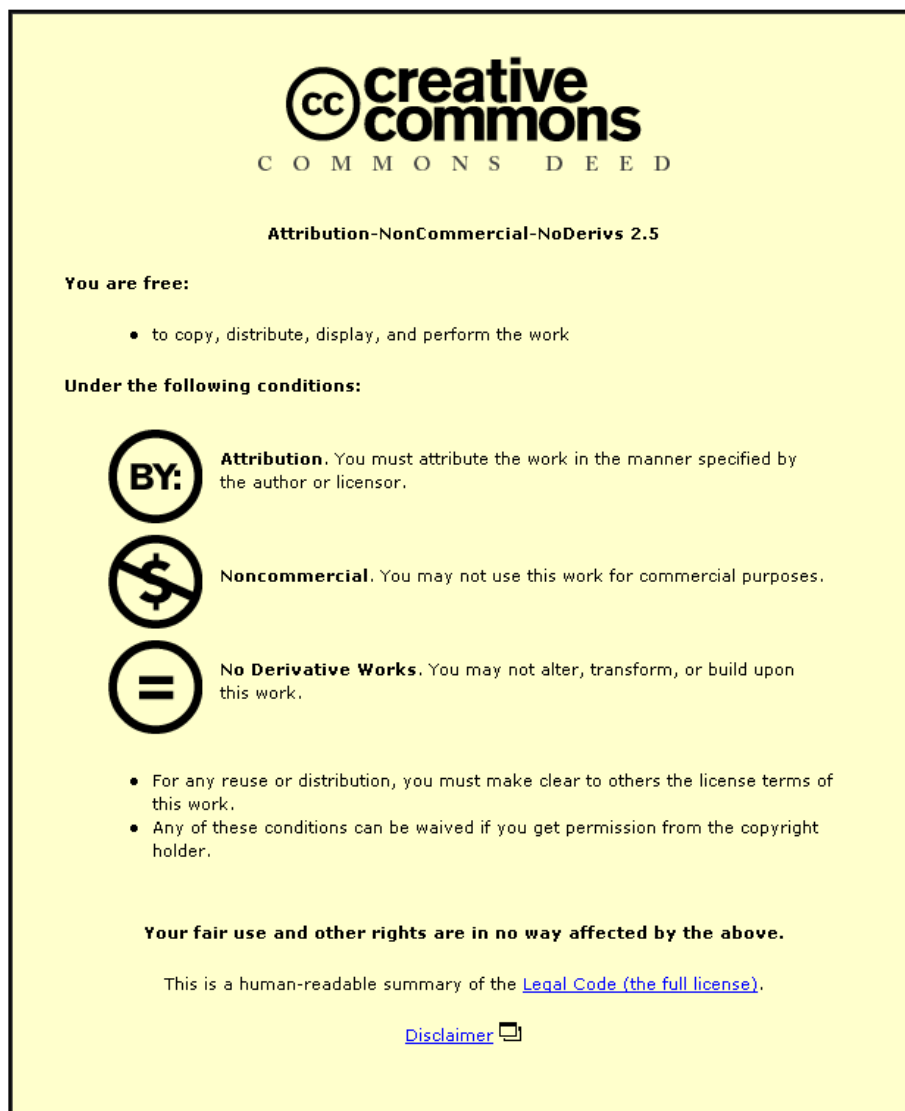


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DETECTION OF VOIDS IN PACKED  
CHROMATOGRAPHIC COLUMNS

by

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A Thesis

Submitted in partial fulfilment of the requirement

for the award of the degree of

Master of Philosophy

of

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## Abstract

The objective of this research has been to develop the understanding of the detection of voids in packed chromatographic columns using one component added to another pure carrier gas. This has been successfully accomplished with an apparatus in which the transients displayed by adding the small gas perturbation flow produced mirror images of the transients displayed by removing the perturbation flow. Flow rates are measured in these experiments. The transients are displayed when a composition front passes a void in the column.

The achievement of the objective is mainly due to the way in which the perturbation to flow is introduced into the system. Instead of the perturbation flow being switched from one column to the other, the flow is simply added and removed sharply from a single column. This has involved the development of a gas sample mixing device which greatly reduces the problem of dead volume. The dead volume causes different effects when the perturbation flow is added and when it is removed. Another aspect of the new system is that the perturbation flow rate can be measured exactly by switching the gas through a soap film bubble flowmeter.

Further development and re-design of the back pressure regulator have successfully eliminated the effect of atmospheric pressure fluctuation on the noise level on the flow rate measurement. Analysis of the deviation in the flowrate caused by desorption and resorption as a composition front passes through a column shows that this research provides, quick, accurate, and a simpler method of obtaining composition residence times from the flow rate records.

## ACKNOWLEDGEMENT

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I would also like to thank Professor B. W. Brooks, Head of the Department of Chemical Engineering, who took keen interest in my progress. Also I wish to record my deep appreciation of the good will and co-operation extended to me by the academic and technical staff, especially Mr L. Moore, as well as the technician T. M. Neale who has helped tremendously. Without them it would not have been possible for me to have completed this work in time.

To my colleagues, whose criticism and discussion have been very useful, I say thanks. They made good company for hard work.

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## CHAPTER ONE

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### 1.0 Introduction

The intent of this chapter is to outline the scope of this research and summarise the history and applications of gas chromatography (GC) techniques. Gas chromatography is a technique in which a separation process takes place in the columns, and which is capable of separating individual components contained in a mixture. By monitoring the composition of the column effluent, information is obtained which can be related to the amounts of the individual components as well as their identification. A modern gas chromatograph is a complex instrument, its installation and proper handling requires both understanding and familiarity to fully optimise the system in order to achieve the best performance.

Although there is much published work on gas-solid chromatography, the application which uses a packed chromatographic column, there is a good deal less on the detection of voids. This is because a void in the column makes little difference (mostly peak spreading) to the outlet composition. A void does however make a difference to the gas flow rate at the column outlet. The fundamental idea of this new work is to show that a void zone in a packed chromatographic column can be detected by measuring perturbation in the outlet flow rate.

### 1.1 Gas Chromatography

The term gas chromatography is applied to all forms of chromatography in which the moving phase is a permanent gas [1]. This technique, which is applicable to volatile substances, may be used with either a solid adsorbent or a liquid as the stationary phase. In this thesis the scope of the work reported here is concerned with gas-solid chromatography, where the carrier (mobile phase) is gas and the stationary phase is solid (5A molecular sieve), packed uniformly into a long narrow glass columns.

In a typical chromatographic application a mixture is added to the upstream end of a column. Once the whole band

is within the column, adsorption - desorption occurs at the front and the rear of the band. During the movement of the mixture along the stationary bed, the separation occurs due to differences in the distribution coefficient of the individual components. The whole process takes place in the column containing the stationary phase through which the moving phase is flowing through the system. At the outlet end of the column, the individual components will emerge more or less separated in time, they are then detected and collected. During the whole process the system is kept at a constant temperature sufficient to maintain the sample in the vapour phase, and to ensure that no decomposition occurs during the process.

## 1.2 Earliest Work of Gas Chromatography

The earliest reported experiments which can be regarded as chromatography are those of Tswett [2] who separated the components of plant pigments by passing their solutions through columns of a solid adsorbent. Following on Tswett's discoveries, nothing further was achieved until, Kuhn, Lederer and Winterstein [3] re-discovered the technique in 1931, although Beri [4] and his associates came near to developing gas chromatography in their attempts at separation by low - temperature sorption and desorption.

The first publications of gas chromatography recognising it as a separate technique were those of Claesson [5] using columns of chemical adsorbent, and somewhat insensitive katharometers as a detectors.

## 1.3 Development of Gas Chromatography

The development of gas chromatography was enormously accelerated in 1952 by the introduction of Gas - Liquid chromatography by James and Martin [6]. Since then the technique has expanded rapidly. After James and Martin's papers, a few workers developed the techniques, until by the middle of 1956 there were several papers on gas-liquid chromatography, which contained most of the basic principles.

Between 1956-1962 most of the present-day instrumentation was established. Since 1962 most attention

has been on instrumental refinements and application of the technique. Recent advances of GC, however, have brought this technology into much widespread use. There have been improvements in sample introduction methods, oven temperature control, detector sensitivity, electrometer stability and speed.

The emergence of flexible fused-silica column technology has been the major factor that has led to its universal acceptance for both special and routine analyses in which high resolution, speed, and sensitivity are required. In addition to the more traditional applications of GC methods it is employed in newer areas such as fine chemicals production, processing of natural gas, and the removal of noxious substances from gases.

#### 1.4 Gas Chromatography (GC) Advantages and Disadvantages

Gas Chromatography (GC) is a unique and very versatile technique. However it requires a high resolution when it is applied to systems containing components with very similar boiling points. Nevertheless by choosing the proper adsorbent, one can separate molecules that are very similar physically and chemically. It is also possible to detect small quantities due to the sensitivity of the thermal conductivity detector (TCD) cells. The TCD can detect a few parts per million; an electron capture detector or phosphorous detector can detect parts per billion and picograms of solute can easily be measured. This level of sensitivity is more impressive when one considers that the sample size used is of the order of  $1\mu\text{L}$  or less. Furthermore, the analysis time of separation of the components in a sample may be reduced to a matter of seconds or minutes. This high separation power is due to:-

- (1) Low viscosity, since the mobile phase has a low viscosity; very long columns with excellent separating power.
- (2) Sensitive detecting systems. Gas chromatographic detectors are relatively simple, highly sensitive and respond rapidly.

- (3) Ease of recording data. The detector output from gas chromatography can be conveniently interfaced with recording potentiometers, integrating systems, computers, and a wide variety of automatic data storing modules.

## 1.5 Uniformity of a packed Chromatographic Column

One of the major advantages of a uniformly packed chromatographic column is that it reduces "peak spreading". A normal composition detector at the column outlet cannot detect the presence of voids. However, if the flow rate of material leaving the column is measured with a sensitive detector (sorption effect chromatography) then any spaces in the peaking can be revealed. This is analysed further Chapter 4.

## 1.6 Separation by Gas Adsorption

Chromatography is an analytical tool but adsorption has become an important unit operation which is employed commercially for the separation of a wide variety of gases. The adsorption of gaseous solutes from air in fixed beds is a basic operation in process technology and is used for purifying air by removing undesirable components, and may also be used for separation of components as in oxygen enrichment.

There are fundamentally two types of gas adsorption processes, which can be differentiated by the way in which adsorbed species are desorbed. In one type the adsorbed species are removed by raising the temperature of the adsorbent and in the other type the total pressure of the system is reduced to effect desorption. They are called temperature-swing and pressure-swing respectively. The first type is used when the solute constitutes only a small portion of the feed gas, while for bulk separations pressure reduction is the preferred mode of separation.

In gas chromatography, the gas separation process is limited in the choice of adsorbent because the amounts to be adsorbed are very small and have little effect. For adsorption separation, the ease of separation is defined as



the separation factor. However the separation factor, which can be based on the adsorbent types, such as Zeolite, depend upon the system temperature and pressure. These quantities are seen to effect the amounts adsorbed.

The phenomenon of adsorption of a solute on a solid surface may be a result of intermolecular forces of the van der Waals [7] type between solid molecules and solute molecules colliding with the solid surface, or result from chemical interaction between the solid surface and the adsorbed component.

The process in the first case is known as physisorption and the latter as chemisorption. The chemisorption process is of fundamental importance in the field of catalysis but is not normally of interest in relation to adsorption as a means of separation for it is often irreversible.

In physical adsorption the gas molecules will tend to attach themselves into the surface of the adsorbent when the intermolecular forces between the solid and the gas phase are greater than those between the gas molecules themselves.

In general terms a dynamic equilibrium is established between the rate at which gas molecules adsorb on the surface and the rate at which they escape to the bulk gas phase. For a given system this equilibrium is determined by both the temperature of the system and the pressure of the gas. Physical adsorption is readily reversible and is accompanied by evolution of heat due to phase change.

The process of adsorption of a gas molecule from a flowing bulk gas stream by a commercial molecular sieve pellet in a bed of adsorbent may be described by the following steps.

- (a) Transfer of the adsorbate from the bulk stream across to the surface of the pellet.
- (b) Transfer of the adsorbate through to the surface of the zeolite crystal.
- (c) Adsorption of the gas molecule onto the surface of a zeolite crystal.

- (d) Penetration by the adsorbate molecule into the intracrystal line cavity of the zeolite crystal.
- (e) Heat release at the surface of crystal due to phase change.
- (f) Transfer of the generated heat to the surface of the pellet by conduction.
- (g) Transfer of the generated heat to the bulk gas stream.
- (h) Transfer of the heat from the bulk gas stream into the wall of the glass tube holding the molecular sieve pellets.

If all the heat transfer rates (items f - h) tend to infinity isothermal adsorption takes place.

### 1.7.a Sorption - effect Chromatography

The sorption effect was first reported by Bosanquet and Morgan in 1957 as the changes in local velocity during gas sorption. Scott (1967) observed both adsorption and desorption peaks using a flame thermocouple detector as a flowmeter. The detector is very sensitive to flowrate changes. However, Buffham, Mason and Co-workers have derived a simple expression to calculate sorption-effect chromatography.

Sorption-effect chromatography differs from conventional chromatography in that it is the flowrate that is measured and not the composition (see section 5.2 page 65).

### 1.7.b Mirror Image Chromatograms

Sorption effect chromatograms can be obtained for the cases of adding and removing a continuous small flow of a component other than the carrier gas. When both of these chromatograms are similar in shape, these are classified as

"mirror-images". In the past experiments the perturbation stream was switched from one side of the system to another, so that both addition and removal effects occurred simultaneously. In the present project, only one side of the apparatus was used thus enabling the mirror-images test to be carried out.

## 1.8 Initial Experimental apparatus

The preliminary experiments were carried out using a packed column to test whether the apparatus was working efficiently to produce the desired results and obtain good quality chromatograms. The initial apparatus set-up used is shown in Figure 1.1 and the experimental details are shown in Table 1.1.

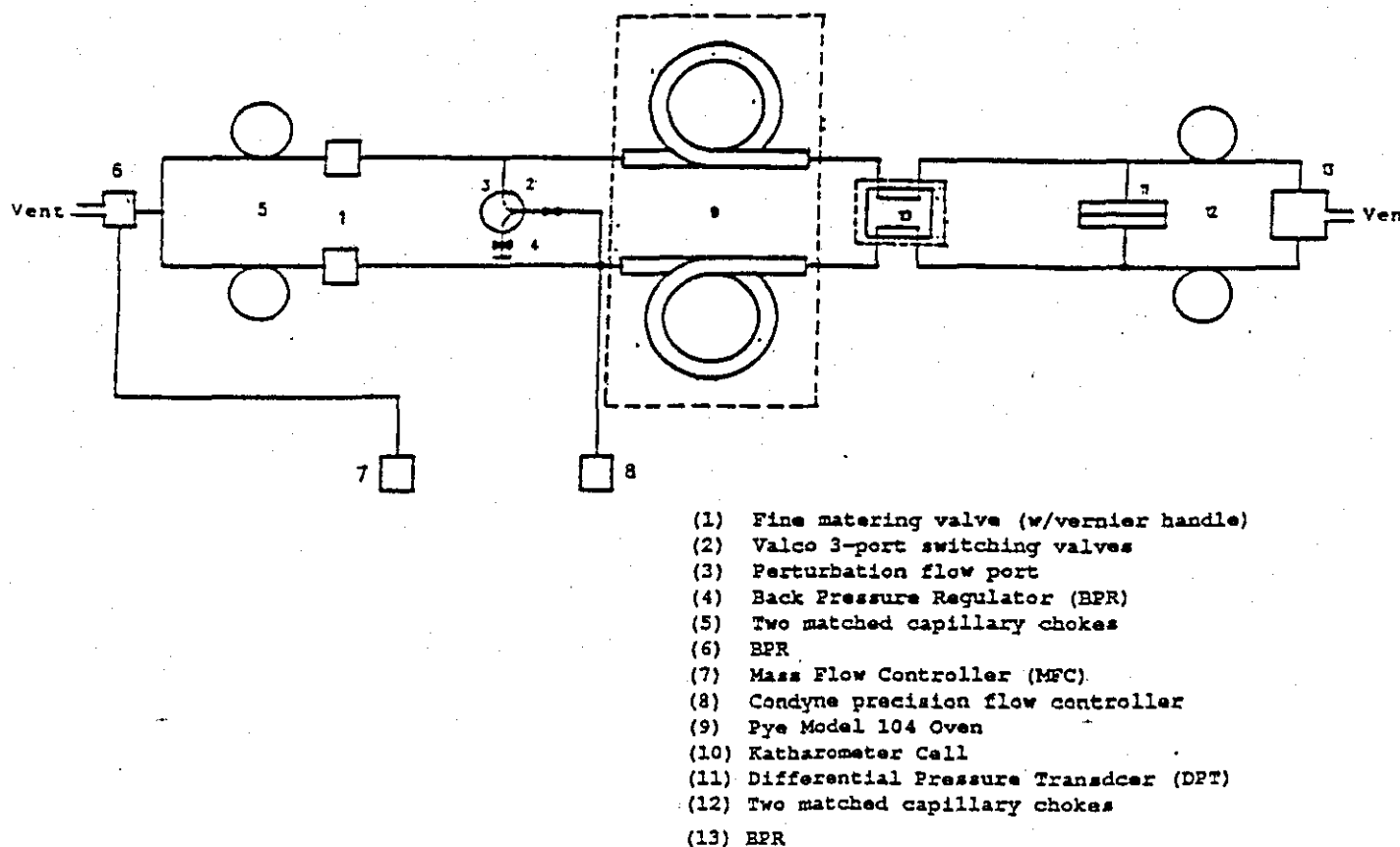


Figure 1.1 Initial apparatus set-up

APPARATUS	DETAILS
Column	Linde 5A molecular sieve (uniformly packed)
System Temperatures	Room Temperatures
Gas Pressure setting	1.25 bar
Carrier Flow-rate	45 mL/min
Chart recorder settings	speed: 20 mm/min sensitivity: 0.2 V

Table 1.1 Details for experiments set-up

A relatively high pressure drop (1-2 bar) across the high resistance chokes and trimming needle valves was used to set constant carrier gas flow rates on both sides of the apparatus. A back pressure regulator kept the upstream pressure constant. A switching valve situated upstream of the columns allowed the perturbation gas streams to be mixed into the carrier flow using a mixing device. The mixture passed through the column. The gas composition at the column outlet was monitored using a thermal conductivity detector.

Capillary flow restrictors were fitted at the column outlet of the apparatus and a differential pressure transducer was connected between the restrictors inlets. A change in flow rate in either capillary is indicated by a change in the differential pressure.

Preliminary experimental runs were carried out at room temperature with a chromatographic column, uniformly packed with Linde 5A molecular sieve. This is important because it showed that the method of adding the perturbation gas produced little or no diffusional spreading. Cutting off the perturbation gas flow should always be a sharp process. On the other hand it is much more difficult to add the perturbation gas without surges or diffusional spreading.

### 1.8.a Initial Results

The chromatograms in Figure 1.2 were obtained using nitrogen perturbation gas and helium carrier gas. The results are shown when the perturbation gas is removed. However, when the perturbation gas was removed from the system the chromatogram shape did not attain the original characteristics. This would suggest that the deviation from the starting point to the point when the front left the measuring capillaries was distorted.

Although the perturbation flow is the same, the viscosity and hence resistance of the column will have changed. This will lead to a change in the flow-rate through one side of the system. Although surely this change is large enough, to be noticed on the chromatogram.

The experiments were repeated this time with a void in the packed chromatographic column. The results obtained are shown in Figure 1.3. The findings suggest that the advancing transients (fronts) are consistent with the previous experiments involving transient separation, but the retreating one is distorted. It seems that when the valve is switched to cut off the perturbation flow, this does not happen immediately.

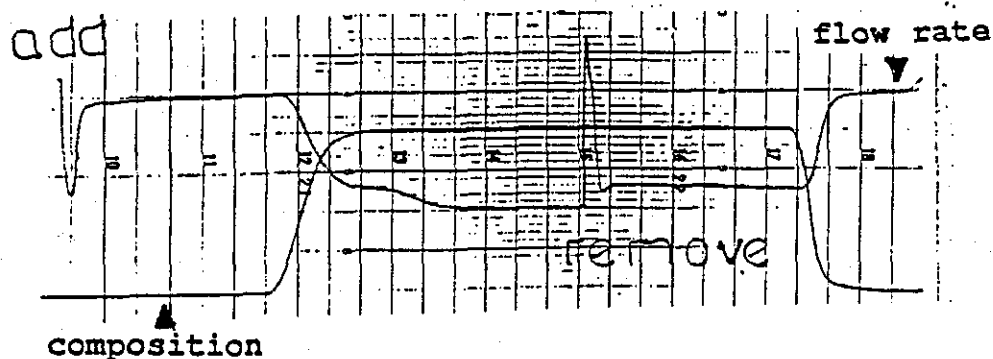


Figure 1.2 Adding nitrogen perturbation gas flow to Helium carrier gas. The chromatograms obtained by addition and removal of nitrogen were not a mirror image.

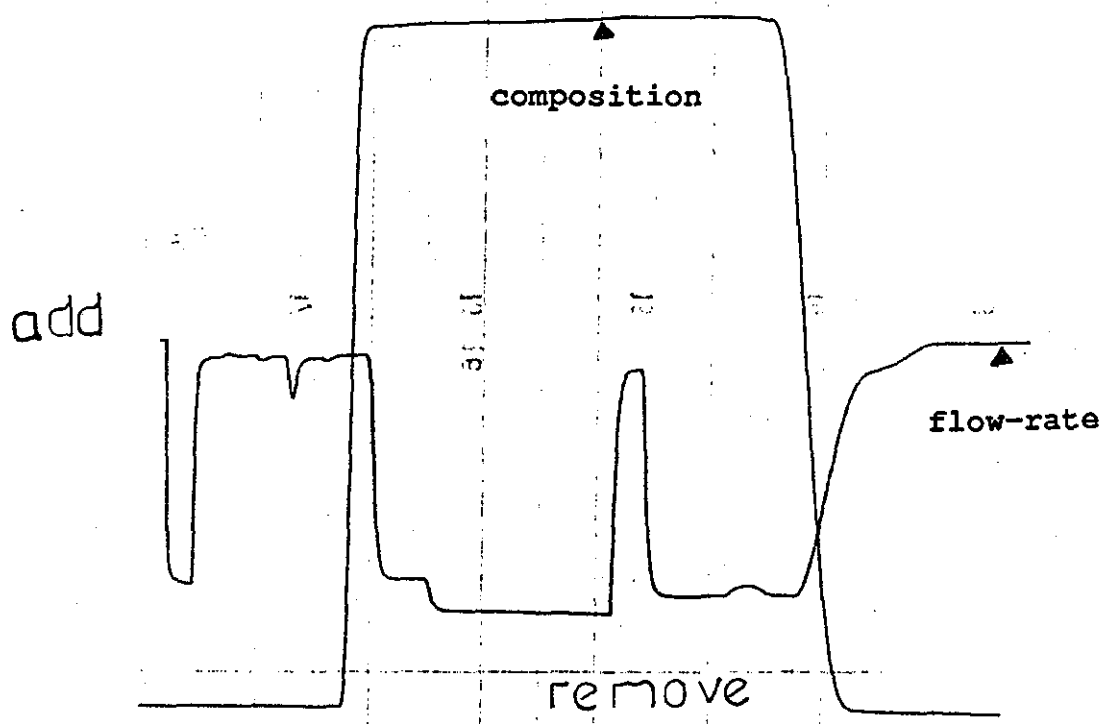


Figure 1.3 Adding nitrogen perturbation gas flow to helium carrier gas. The column contained a void. The chromatograms obtained are not a mirror-images.

The problem is the amount of dead volume. After the perturbation gas flow was switched off some of the gas continues to diffuse out from the dead volume into the system. In the experiments, the dead volume causes the response when adding the perturbation flow to be different to the response when cutting the flow off. If the responses are mirror images of one another, then the dead volume is so small as to have no effect.

From the initial results, the experimental arrangement was not sufficient to obtain mirror image transients for both adding and removing the perturbation gas flow. This also, can be clearly seen when nitrogen perturbation is added to argon carrier gas, Figure 1.4. From these results it was evident that the experimental arrangement required modification in order to eliminate any diffusional effects and hence obtain mirror image of the transients.

These diffusional effect are prevalent when the perturbation flow is removed from the carrier gas. Because of the diffusional effects, the peak obtained previously

in Figure 1.3 shown as the front passes through the void will be flattened and the area of this void will be difficult to determine.

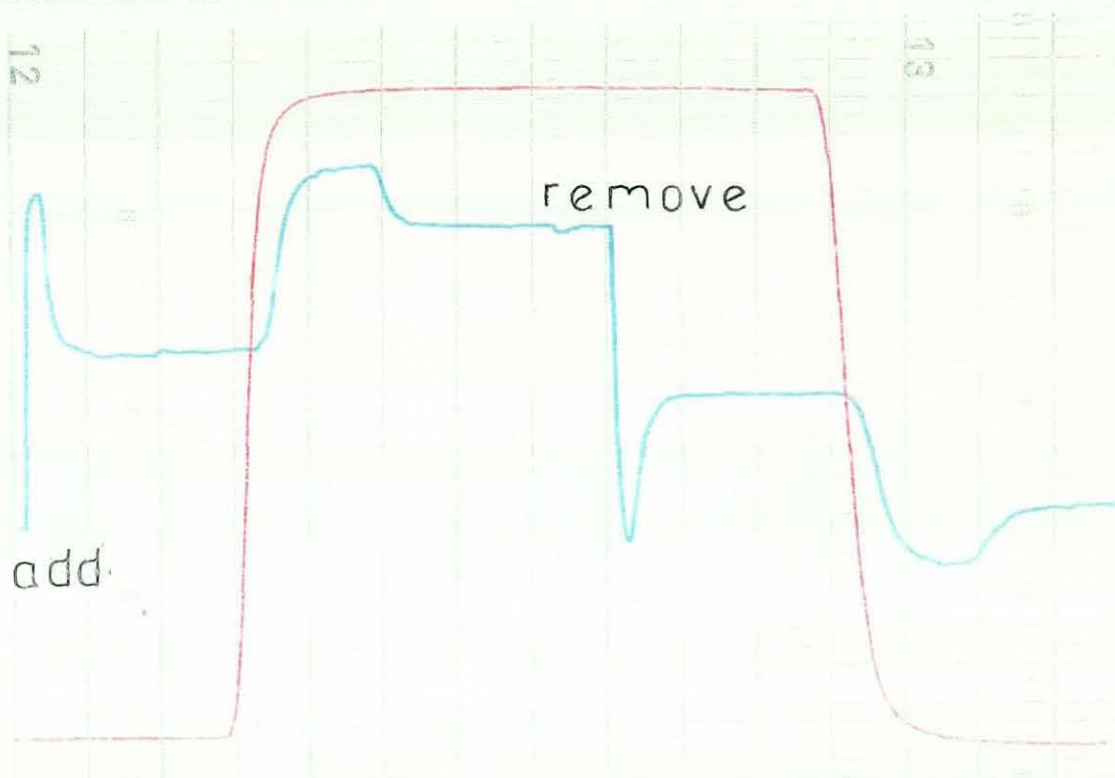


Figure 1.4 Shows 0.4% of nitrogen the perturbation gas flow with argon carrier. No mirror image was obtained.

### 1.9 Experimental Arrangement Modification

From the results of the preliminary experimental arrangement, modification is required to reduce this dead volume as much as possible, and to improve the chromatogram quality (refer to Chapter 3).

The initial experiments were carried out without upstream delay lines (a section of empty tube). In a second set of experiments a delay line was added to the apparatus (Figure 1.5) thus separating the flow rate change caused by desorption from the pressure change caused by the viscosity in the capillary flow meter changing.

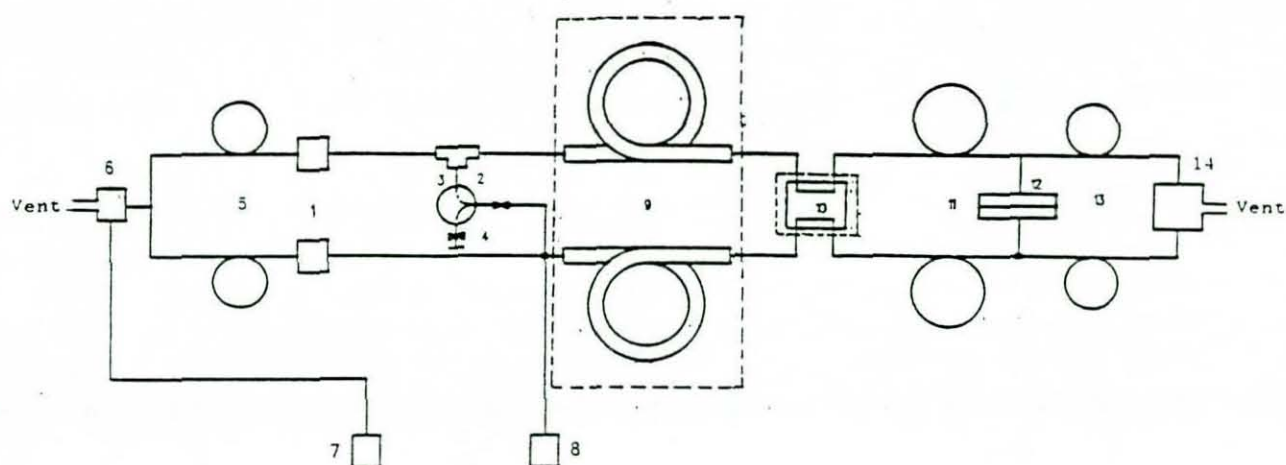
Delay lines are simply low pressure-drop tubing which delay the passage of composition fronts through the system.



Their advantage is that they allow separation of events. For example, the upstream delay lines allow separation of the addition of a small perturbation flow and its arrival (and adsorption) at the column. Delay lines are conventionally used downstream of the column and upstream of the flowmeter. The flowmeter measures the column flow-rate by monitoring the pressures upstream of the measuring capillaries. However, this pressure will also be affected by the viscosity as well as the flow-rate.

The viscosity will vary with gas composition. Thus, in order to monitor the flow as the composition front leaves the column, delay lines placed upstream of the flowmeter are needed to ensure that the gas composition in the capillaries remains constant for the time the front is leaving the column. Some time later the composition front arrives at the capillary tubes of the flowmeter and a further deflection will be observed. As well as the upstream delay line an additional 3 meter delay line length of 1/8 inch tubing was fitted downstream of the column, so as to detect the steps of the flow rate on the chart recorder without the effects of viscosity. In order to obtain reasonably large sharp sorption effects on a smooth baseline, it is necessary to choose a non-sorbed carrier gas and a modestly sorbed perturbation species such as helium as carrier, and nitrogen as the perturbation gas.





- (1) Fine maturing valve (w/vernier handle)
- (2) Valco 3-port switching valves
- (3) Trace Mixing Device (TMD)
- (4) Back Pressure Regulator (BPR)
- (5) Two matched capillary chokes
- (6) BPR
- (7) Mass Flow Controller (MFC)
- (8) Condyna precision flow controller
- (9) Pye Model 104 Oven
- (10) Katharometer Cell
- (11) 10 meters of Nylon tubing (delay line)
- (12) Differential Pressure Transducer (DPT)
- (13) Two matched capillary chokes
- (14) BPR

Figure 1.5 Apparatus second set-up

## 1.10 Preliminary Experiments Results

The results obtained will be divided into two sections corresponding to each system. There will be a few sentences of description. Firstly, the system was set up with two identical columns and the perturbation flow was switched from one column to another producing chromatograms in Figures 1.6 - 1.8. Secondly the system was set up with additional identical delay lines added upstream of the column. Only one column was used, and the perturbation flow was either added or removed from the particular column. The resulting chromatograms are contained in Figures 1.9 - 1.10.

### 1.10.1 Results Obtained Without Upstream Delay Line

- (a) Adding nitrogen perturbation gas flow to helium carrier flow. Figure 1.6 shows what happens when a small flow of nitrogen is added to the helium carrier gas. The nitrogen flow rate was 0.3% of the flow of helium the upstream gas pressure was 1.25 bar, the chart recorder speed was set at 30 mm/minute and with a sensitivity of 0.2 volts. The graphs correspond to when the nitrogen is added to the system.
- (b) Adding helium perturbation gas to argon carrier gas. Figure 1.7 is the chromatogram resulting from adding a small flow (0.5% of the total carrier flow) of helium to an argon carrier flow. The chromatograms for addition and removal are mirror images.
- (c) Using nitrogen perturbation gas with argon carrier gas. Figure 1.8 shows a typical response when a small nitrogen flow (0.7% of the total carrier flow) is added to an argon carrier. Again mirror-image chromatograms are achieved.

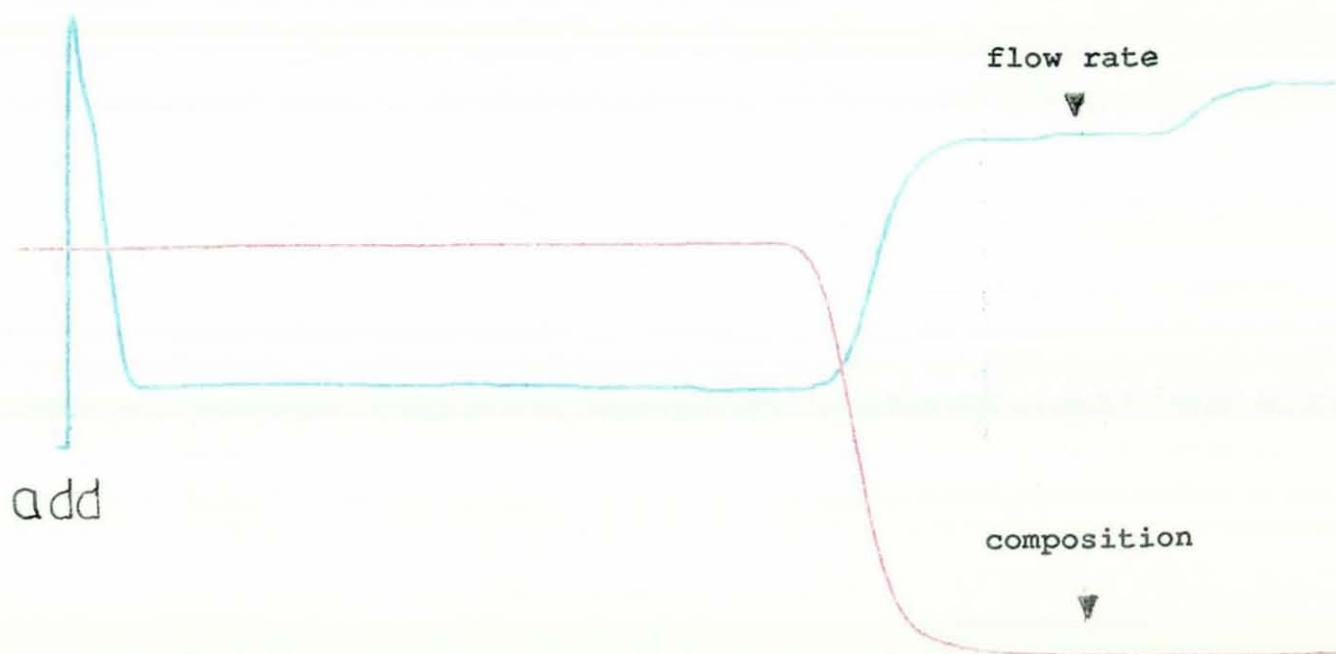


Figure 1.6 chromatogram shows nitrogen perturbation flow added to helium carrier gas flow

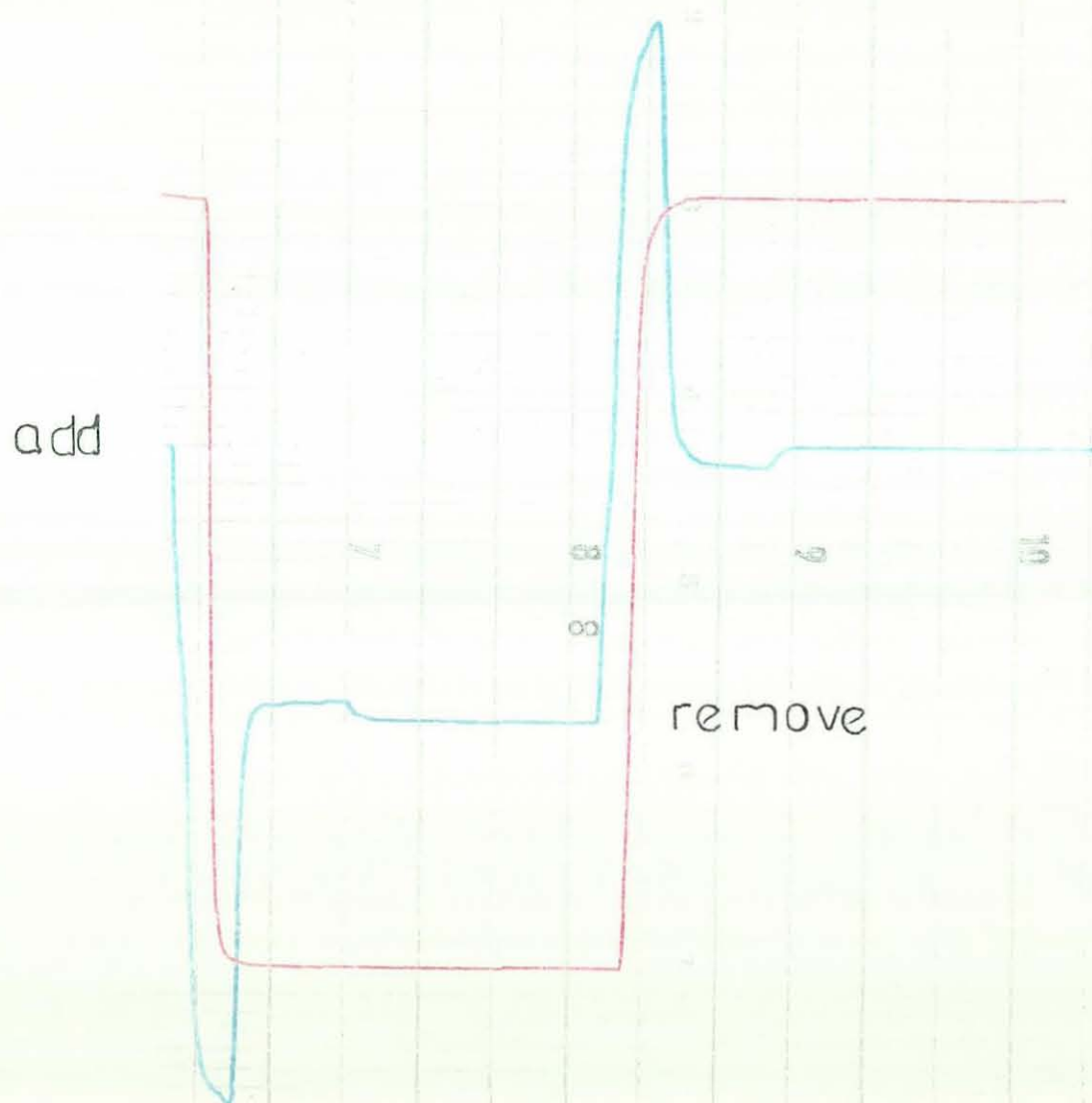


Figure 1.7 mirror image of chromatogram for a 0.5% Helium perturbation flow is added to argon carrier gas.

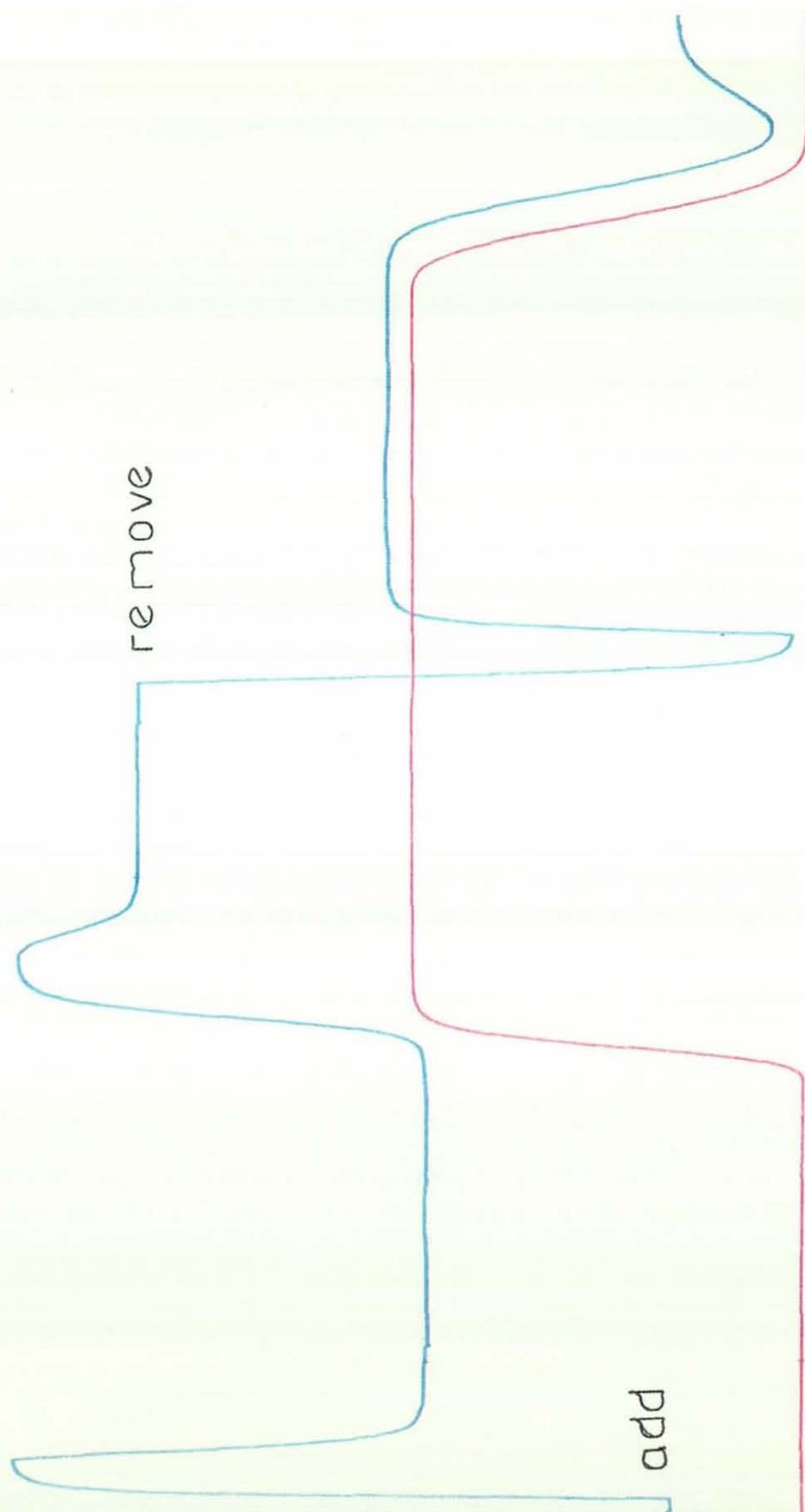
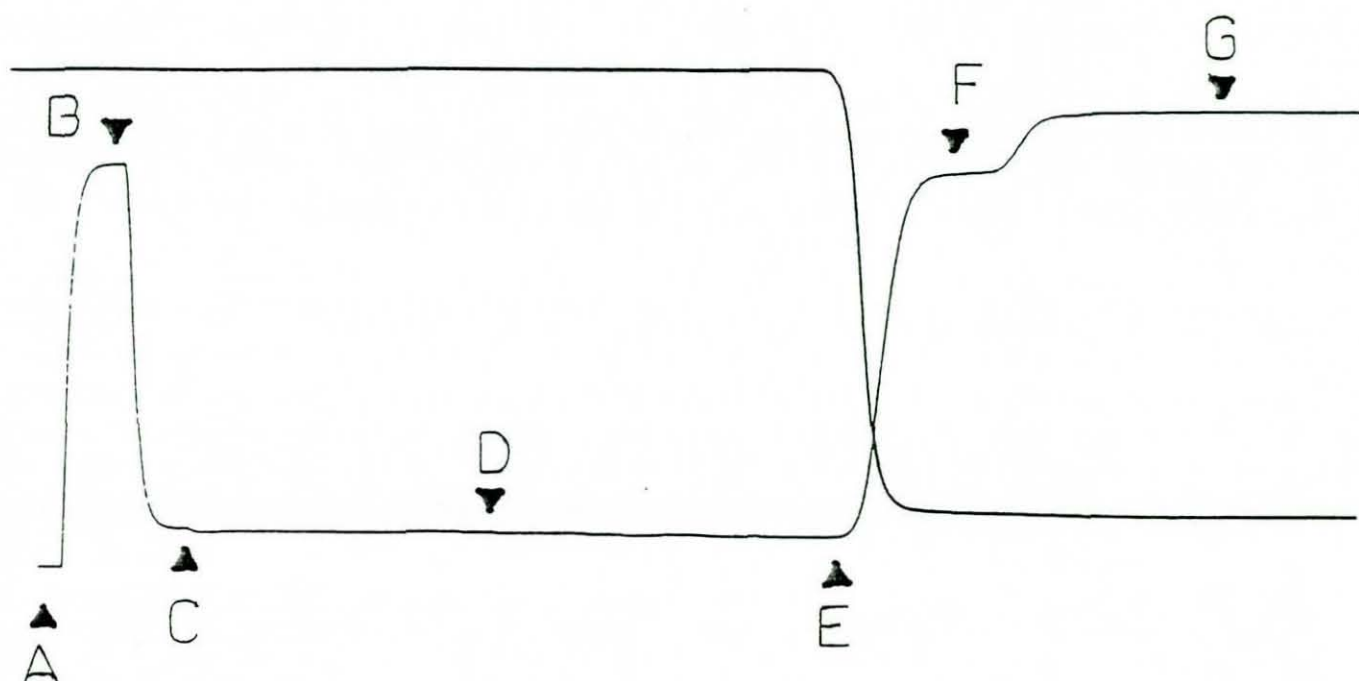


Figure 1.8 Mirror image chromatogram for a 0.7% nitrogen perturbation flow added to argon carrier.



## 1.10.2 Results Obtained With Delay Line

- (a) Adding nitrogen perturbation to helium flows. Figure 1.9.a. gives a schematic representation of 0.3% of nitrogen perturbation gas to the flow rate transients (helium). Because the composition front is reasonably sharp, a plateau is obtained at position F, before the composition front leaves the downstream delay line and enters the measuring capillaries.
- (b) Figure 1.9.b shows what happens when a small flow of nitrogen is added to the helium carrier gas. The nitrogen flow rate was 0.3% of the helium carrier. The upstream pressure was 1.25 bar and the chart recorder speed was set at 20 mm/min and with a sensitivity of 0.5 volts. The graphs produced when the nitrogen is added to the system and when it is removed, as shown in Figure 1.9.c are mirror images of each other.
- (c) Using argon perturbation gas with helium carrier gas. Figure 1.10 shows the chromatogram resulted from adding a small (0.5% of the total flow) flow of argon to a pure helium carrier. The chart recorder speed was 20 mm/min, the same as Figure 1.6, and the sensitivity was also 0.2 volts. The chromatogram for removal of argon was a mirror image of the chromatogram for addition.



- A: adding trace
- B: front moving through upstream delay lines
- C: front reaches column
- D: front in the column
- E: front leaves the column
- F: front moving through downstream delay lines
- G: front through the measuring capillaries

Figure 1.9 A Schematic chromatogram obtained for adding a 0.3% nitrogen perturbation flow to helium carrier gas. It also show different stage of the system behaviour.

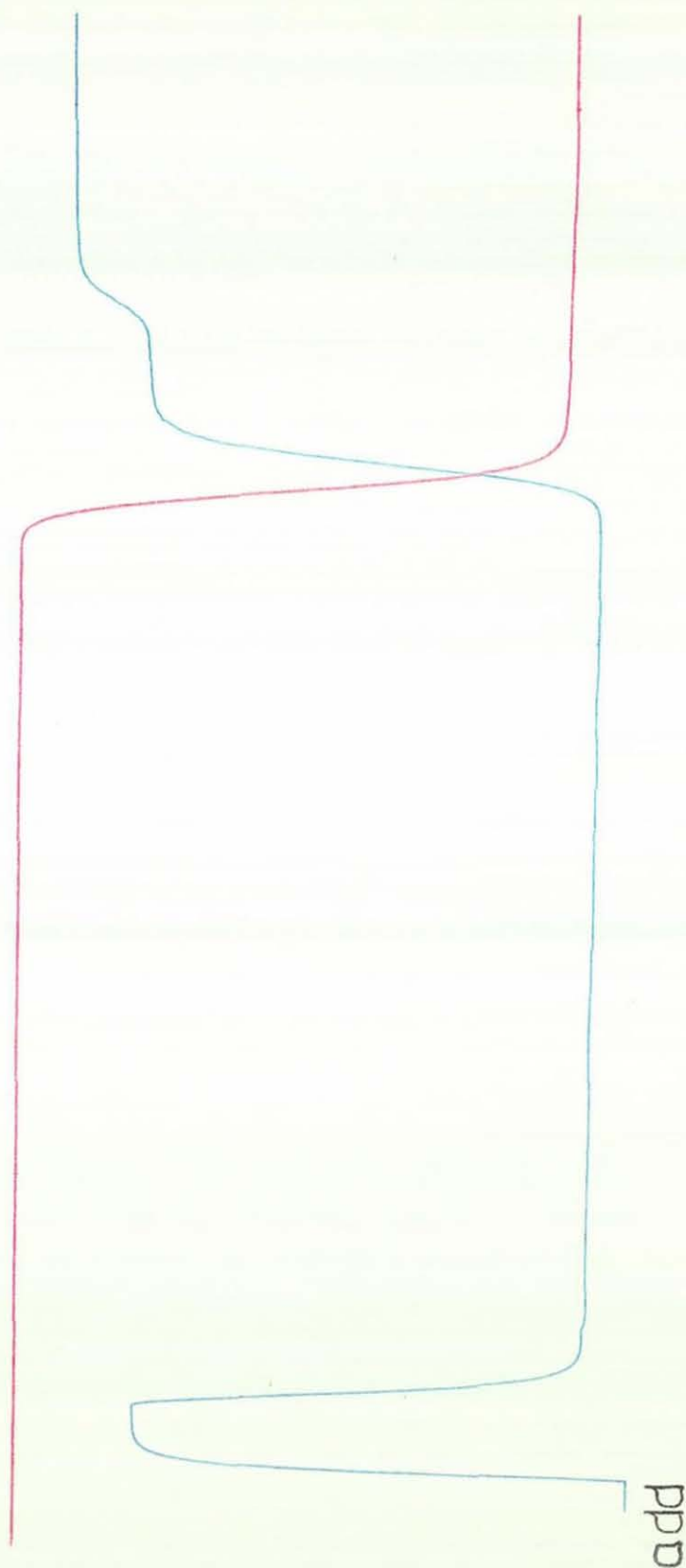


Figure 1.9 B Illustrating the graph obtained when adding a 0.3% nitrogen perturbation flow to helium carrier gas.



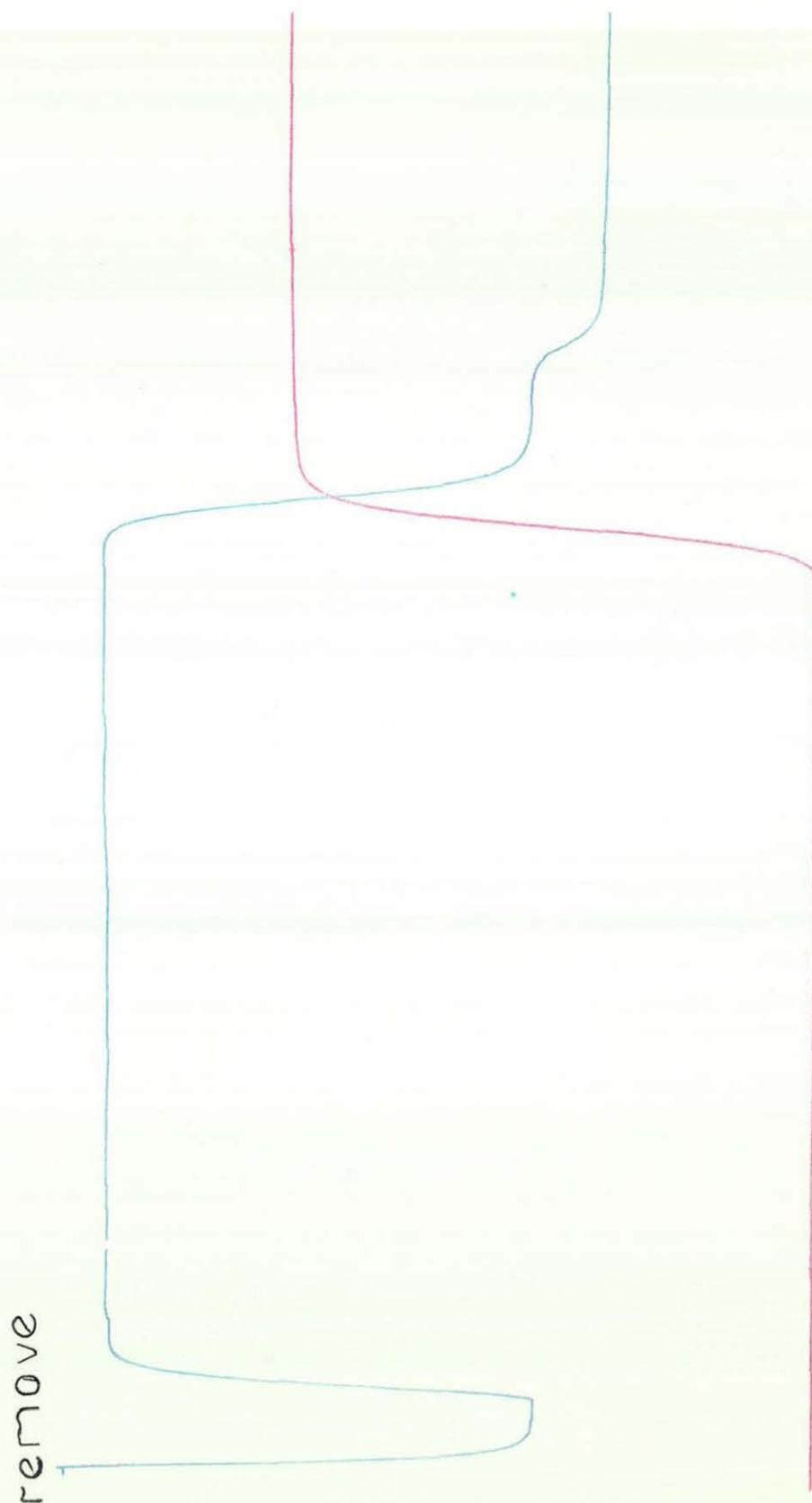


Figure 1.9 C show the graph obtained when 0.3% of perturbation nitrogen flow is removed from the system.



Figure 1.10 Shows the chromatogram that resulted from adding a 0.5% Argon perturbation flow to helium carrier gas.

## 1.11 Summary of Preliminary Results

Initially, experiments were conducted by switching a perturbation flow from one column to the other using a six port valve, and measuring the cumulative effect. Leakage problems were evident and so it was decided to employ a three-port valve instead. The results have revealed that using one column, a mixing device and a three port valve, mirror images are obtained, of the transients corresponding to adding and removing the perturbation flow.

- (a) From the preliminary results, the apparatus suffers from random fluctuations in atmospheric pressure. This was seen to be a potential problem, and so modifications to the apparatus were required to reduce noise level.
- (b) The use of upstream and downstream delay-lines has shown that the plateau is properly attained when the composition front leaves the column and passes into the flowmeter. This helps to obtain the offset required for void area determination. Also, the use of upstream delay lines enables the area required for the flow rate retention time determination, to be clearly defined.
- (c) Modifying the system reduced the dead volume and the sensitivity increased. This meant that a relatively small change in the flow-rate can be detected.

## 1.12 Research Objectives

The work described in this theses begun in mid-1989 when the author was working in the Department of Chemical Engineering, Loughborough University of Technology.

The aim of the work was to investigate the detection of voids in packed chromatographic columns. Traditionally a perturbation gas has been switched from one column to another resulting in cumulative effects of advancing and retreating fronts. The present work, technique (developed by Mason and Buffham) is a considerable simplification over the previous method. One

packed column is used and the flow of perturbation gas is switched from column to vent and vice-versa.

#### 1.12.1 Experimental Programme

- (a) To add nitrogen perturbation gas to helium carrier gas in a column with a void to see if the flowrate records are similar in shape.
- (b) To assess the similarity in shape of the chromatograms using different combinations of gases and columns.
- (c) Normally the composition residence time has to be obtained using a katharometer but it should be possible to obtain it from the flow rate trace. This requires investigation by removing the upstream delay line and thus eliminating the residence time associated with it.
- (d) To calculate the composition residence time,  $T_x$ , and the flow rate residence time,  $T_n$ , using simple procedures.
- (e) To calculate the composition residence time from the flow rate.
- (f) To calculate binary adsorption isotherms for argon - nitrogen using the flow-rate trace only.

## CHAPTER TWO

### MOLECULAR SIEVES [ZEOLITE]

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#### 2.1 Zeolite

Molecular sieve zeolite 5A is used in industrial applications to separate air into oxygen and nitrogen. Many gas<sup>es</sup> contain other impurities, in addition to water, which can be simultaneously adsorbed by molecular sieves. For this research, chromatographic columns are packed with, molecular sieve zeolite 5A. The preliminary experimental runs were carried out using different sizes of molecular sieve. The results show that the chromatographs are affected by the size of the particles.

The name zeolite is derived from a Greek word meaning to boil, and it refers to the swelling-up and apparent boiling that occurs when these minerals are heated and water is driven off.

#### 2.2 Characteristics of Molecular Sieves

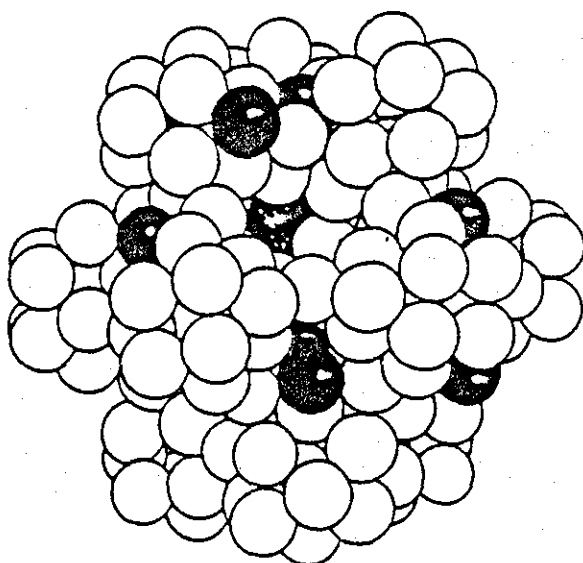
The zeolites form a large family of aluminosilicates and have been studied by mineralogists for more than 200 years [8]. The three industrial useful properties of zeolites which are: their capacities to sorb gases, vapours and liquids; to catalyse reactions; and to act as cation exchangers.

Zeolites are crystalline hydrated alumina silicates of sodium, potassium, magnesium, calcium, strontium and barium. When the water of hydration is driven off the crystal does not collapse or rearrange but rather gives cavities which are interconnected by empty pores of fixed and uniform dimension varying from about 3 - 10 Angstrom units in diameter (see Figure 2.1). Structurally the zeolites are "framework" aluminosilicates which are based on an infinitely extending three-dimensional network of  $AlO_4$  and  $SiO_4$  tetrahedra linked to each other by sharing oxygen atoms.

The molecular sieving properties of zeolites are uniquely determined by their pore diameters, the magnitude of which determines which sized molecules will be totally excluded from the interior of the zeolite.

Quite apart from their molecular sieving effects zeolites are also effective in selectively adsorbing particular components from a mixture of molecules capable of penetrating the entire zeolite. This is caused by the interaction of the zeolite with certain molecular properties such as the dipole moment. One commercial selective adsorption process is the separation of oxygen from air by the preferred adsorption of nitrogen. Another is the selective adsorption of sulphur compounds from natural gas. A third is the selective adsorption of aromatics from hydrocarbon mixtures.

# *Linde*



## MOLECULAR SIEVES

Fig.2.1 shows the Linde molecular sieves pores structure.

Compared with other adsorbents such as silica gel, activated alumina and activated carbon, molecular sieve adsorbents can offer higher adsorption design loadings, resistance to fouling and coking, more complicated impurity removal from the process stream and unique selective adsorption based on molecular size. In commercial operations these properties may lead to reduced adsorbent requirements, lower pressure drop, greater operating flexibility, elimination or reduction of product loss due to coadsorption, longer adsorbent life and more reliable and uniform performance.

The molecular sieves which are of interest in air separation are type A, type X and mordenite-type. For types A and X, they are normally produced as 4A (NaA) and 13X (NaX) in which sodium is the alkali metal. Through an ion exchange mechanism about 75 per cent of the sodium ions are replaced by calcium ions thus producing type 5A (CaA) and 10X (CaX). The effective pore diameters of all of these types are shown in Table 2.1 and the kinetic diameters for various gases are given in Table 2.2.

In order to utilise the adsorption characteristics of zeolites in separation processes, commercial molecular sieve adsorbents are prepared as pelleted agglomerates containing a high percentage of the crystalline zeolite together with the necessary amount of an inert binder amounting to about 20% of the total weight of the pellet.

Adsorption equilibrium in molecular sieves has been extensively studied and the behaviour of systems involving a single adsorbable component is now fairly well understood. However, the practical applications of zeolites as selective adsorbents generally involves the adsorption of multi-component mixtures. Available experimental data for such system are very limited.

Other work has been presented on type 5A molecular sieve, and also a number of techniques for predicting binary equilibria adsorption from pure component isotherms have been published. The lack of a comparative analysis of their merits due to lack of experimental data makes it difficult to choose between one particular zeolite and another.

Diameter A° Type		Nominal Pore
4A		4
5A		5
13X		10
10X		8
Na-Mordenite		6

Table 2.1 Nominal Pore Diameter for Different Zeolites

Gas	Kinetic Diameter A°
He	2.60
H <sub>2</sub>	2.89
Ar	3.40
O <sub>2</sub>	3.46
N <sub>2</sub>	3.64
CO	3.76
CO <sub>2</sub>	3.30
SO <sub>2</sub>	3.60
H <sub>2</sub> S	3.60
CH <sub>4</sub>	3.80
C <sub>2</sub> H <sub>2</sub>	3.30
C <sub>2</sub> H <sub>4</sub>	3.90
C <sub>3</sub> H <sub>2</sub>	4.30
Cyclopropane	4.23
Propylene	4.50
Benzene	5.85
Cyclohexane	6.00

Table 2.2 Kinetic Diameters for Various Gases.



## CHAPTER THREE

# APPARATUS AND EXPERIMENTAL WORK

---

### 3.1 INTRODUCTION

This chapter describes the experimental apparatus and the procedure used for the detection of voids in packed chromatographic columns using different combination of gases; Nitrogen, Helium, and Argon.

The materials employed for packing the columns and creating the voids were Linde 5A molecular sieve and glass ballotini. The experimental runs were carried out at different temperatures, to assess the effect of voids on the flow rate behaviour. Although there were two sides of the apparatus, perturbations were only made to one side. The other side provided a dynamic balance for the "active" side.

Normally a composition retention time is obtained by using a katharometer and interpreting the record. The retention time can also be obtained from just the perturbation flow. However, this requires the apparatus to be operated without the upstream delay lines because otherwise, the presence of the upstream delay lines would give an unrealistically large value of the composition retention time. Alternatively the residence time in the delay line can be subtracted from the composition residence time.

### 3.2 Experimental outline

The basic apparatus and the method used during the experimental work was different from previous apparatus set-ups [9 and 10]. This due to the change in the way the small flow of perturbation gas was introduced to the system.

Instead of the small perturbation flow being switched from one column to the other, the flow was simply added and removed from a single column. This required the

Effectively, with a dead volume present the perturbation flow is not cut off sharply because of the presence of gas which slowly diffuses out from the dead volume. Adding perturbation flow can be done sharply. In the method which had been used previously, one gas was replaced by another and the dead volume was not as much of a problem. Another aspect of the new system is that the perturbation flow can be measured by switching the flow to vent and measuring the flow directly with a pipette and soap film bubble flowmeter. Moreover, with the new system, very small perturbation flows of down to 0.1 ml/min can be obtained and measured precisely. With typical bulk flows of 25 ml/mins, the perturbation flow can be as small as 0.4 % of the bulk-flow.

Another new introduction is the interfacing of a differential input/output data acquisition (MIN - 16) card to an IBM PC Computer. The card occupied into one of the peripheral slots inside the computer, and could receive the output voltage from the DPT and convert it into an electrical signal. This communication link provided real time processing.

The interfacing of 4-wire platinum Resistance Temperature Detector (RTD) to the computer to monitor column oven temperature and the katharometer cell temperature was also established. Essentially the system consists of identical columns packed with Linde 5A molecular sieve set in parallel. One contains the void to be identified and measured, the other has no voids. Downstream from the columns there is a sensitive differential flow meter incorporating a differential pressure transducer. To ensure equal flows through the columns, two matched capillary chokes were placed upstream of the columns, being connected to a further pressure regulator. A switching valve was attached to the head of the columns to introduce the sample gases to the system. Figure 3.1 illustrates the apparatus layout.

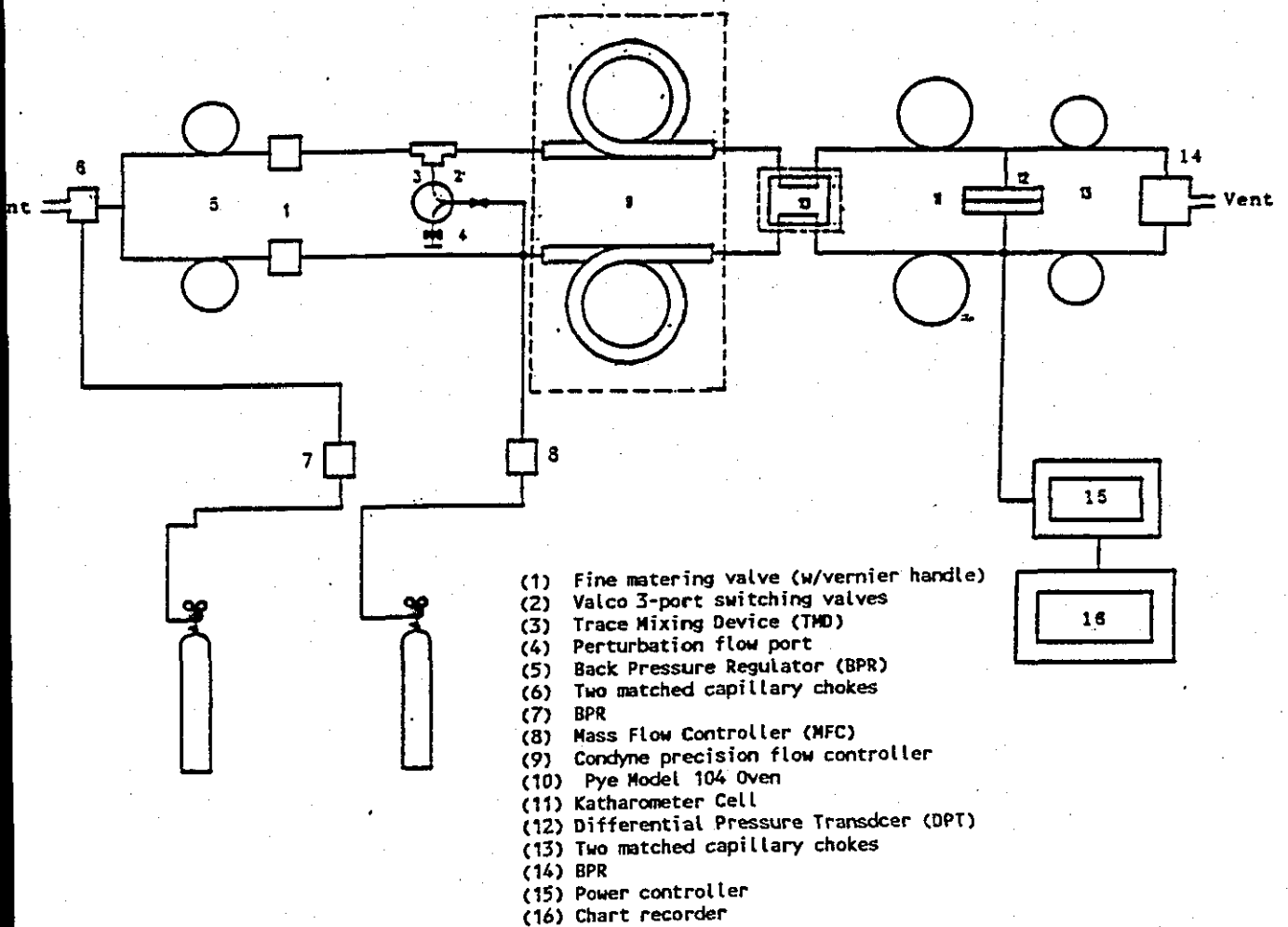


Figure 3.1 Illustrating The Overall Apparatus Configuration.

### 3.3 Apparatus Descriptions

#### 3.3.1 Pye Series 104 Chromatographic Oven

The Pye 104 oven can be used to provide accurate column temperature between 35 and 500°C although in the present project experiments were carried out at room temperature and at 60°C. The accompanying equipment allows programmable temperature runs, involving ramp changes between pre-set initial and final temperatures. The experimental runs in the present work require a constant temperature.

The oven door, which opens downwards, may be latched in the 45° position. This allows hot air from the oven to be deflected upwards if rapid cooling is required between runs. The column temperature is sensed by a platinum resistance thermometer which is supported in a stainless steel half-cylinder and mounted from the back of the oven.

A thermal fuse, fitted in the top right-hand corner of the oven compartment, provided protection against excess temperature. The heater element is of low thermal mass and surrounds a centrifugal fan driven by an electric motor which is mounted in the space behind the oven compartment.

The air circulation (Fan) ensures efficient heat transfer between heater and column and even temperature distribution through out the oven more quickly. Also, the rate of cooling of the oven when the heater has been switched off is increased by use of the high fan speed. Figure 3.2 shows the oven and a position of the oven fan. For isothermal runs at oven temperatures below 50°C, and when a reduction in mechanical noise is required, low fan speed is recommended.

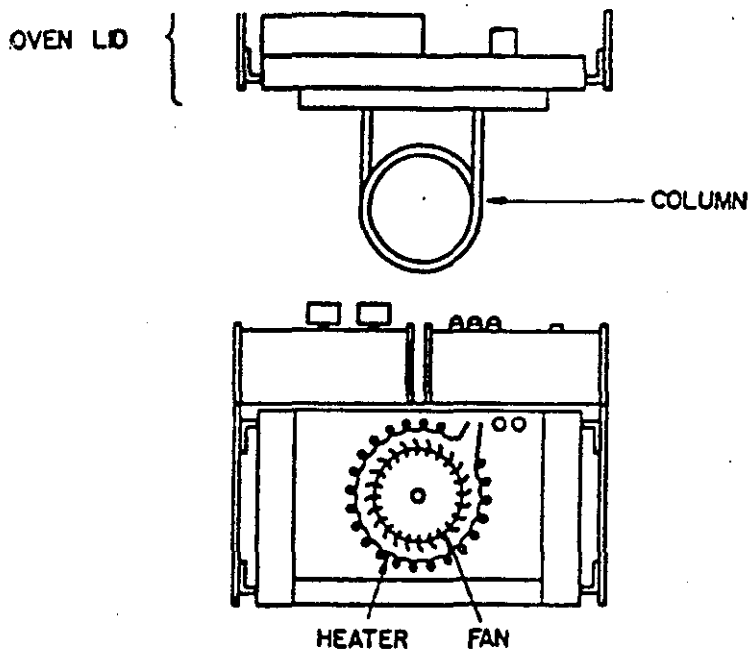


Figure 3.2 - Front View of the Pye 104 Oven

### 3.3.2 Thermal Conductivity Detector (TCD) Cell

The thermal conductivity detector is the most commonly used measuring device in gas chromatography. It senses the amount of each substance separated by the column. Figure 3.3.a shows the thermal conductivity cell, which measures the small change in thermal conductivity of the carrier gas caused by the presence of the eluted substances.

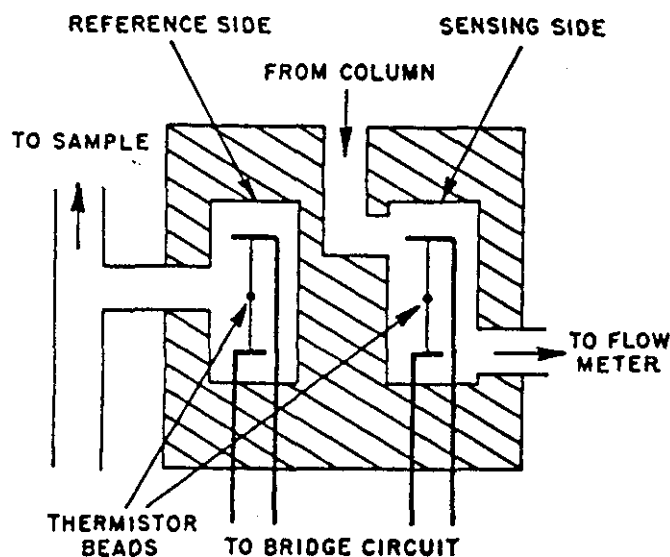


Figure 3.3.a The Thermal Conductivity Cell

In most arrangements, the Katharometer or TCD is situated in the oven. However, in the present arrangement it is attached out side the oven and connected to the power supply model 40-202 as shown in Figure 3.3.b. It was surrounded by an electric blanket heater and this in turn had an asbestos block around it.

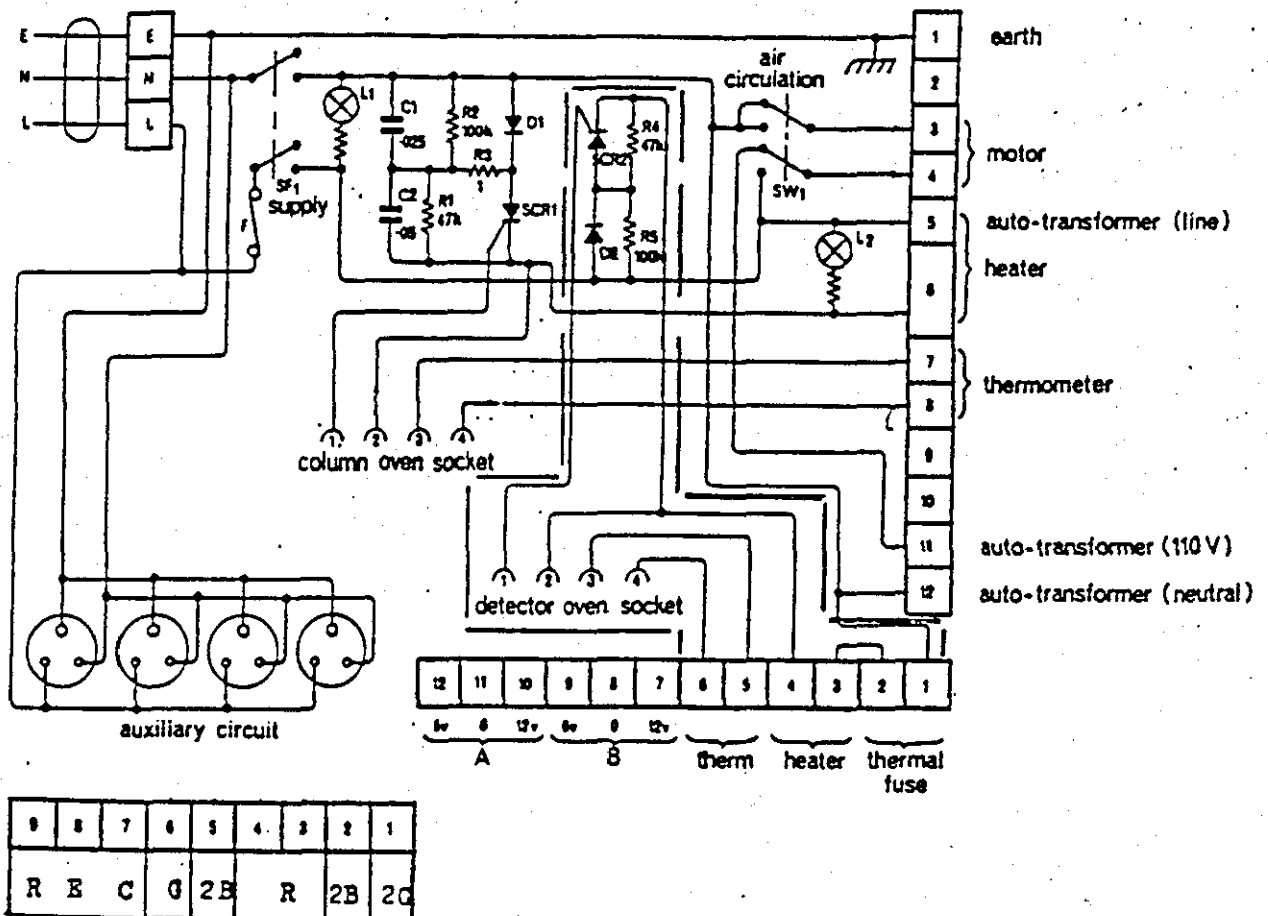


Figure 3.3.b Circuit Diagram of Power Supply to the Katharometer Cell

The name katharometer was introduced by Shakespeare 1958 [11]. The katharometer design is robust, very simple and is relatively cheap, it is also easy to operate for sensitive analytical work. However, thermal conductivity detection system are based on two principles:

- (1) Each gas has its own thermal conductivity, and for a binary mixture this thermal conductivity will change with composition.
- (2) The metal filaments and thermistors (semiconductors of fused metal oxides) have fixed resistance temperature relationships.

This device consists of a single metal block through which two cylindrical chambers have been cut to form a pair of thermal conductivity cells. The use of a single metal block ensures that the walls of both cells are at the same temperature. Each cell consists of a platinum wire mounted coaxial within the cylindrical chamber to form two of the arms of a Wheatstone bridge network.

The gas flows through the chamber and the wires are heated by a closely controlled electric current to a temperature which depends on the gas flow and the thermal conductivity of the gas passing over it.

The temperature of each wire, and hence its resistance, varies with the thermal conductivity of the surrounding gas. Table 3.1 shows typical values of thermal conductivities measured relative to nitrogen. These include the three gases used in the experiments.

Gas	0°C	100°C
Helium	5.89	5.54
Nitrogen	1.00	1.00
Argon	0.68	0.71

Table 3.1 Relative Thermal Conductivities of Gases used.

In practice, there are four cells connected in a bridge circuit. Carrier gas flows through one pair and the gas containing the mixture flows through the other pair. Gas flowing through the channels conducts heat to the wall because the wall, always has a lower temperature.

However, if a binary mixture emerges from the column, the sensing element will have a new temperature, because the thermal conductivity of the gas stream and consequently the rate of heat conduction have changed. Because the wall temperature remains constant, the temperature (and resistance) of the element must change. The effect of this resistance change is measured and converted into small e.m.f which can be recorded. If pure carrier gas flows through the channels and the resistance of each element is the same and then they are balanced. When a foreign component appears in the measuring channel, the temperature and resistance of the element will change and deflect the balance on the bridge(out of equilibrium). This deflection is plotted by the recorder connected to the bridge as a zero instrument.

The thermal conductivity of pure gases can be defined in relation to kinetic theory as :

$$K = 1/3(\overline{mnc}C_v) \quad (3.1)$$

where

$K$  = Thermal conductivity of the gas (quantity of heat in ergs flowing per second through  $1 \text{ cm}^2$  temperature gradient of  $1^\circ \text{ C /cm}$  ).

$m$  = Mass of each molecule

$n$  = Number of molecules in 1 ml of gas

$c$  = Average or mean molecular velocity

$l$  = Average or mean free path

$C_v$  = Specific heat of the gas at constant volume

In turn,  $K$  bears a relationship to the coefficient of viscosity,  $\eta$ , which is equal to  $1/3(\overline{mnc}l)$  or  $1/3(\rho cl)$ , where  $\rho$  is the density or mass of unit volume,  $\text{mn}$ .



Therefore, thermal conductivity can be defined as:

$$K = 1/3(\bar{q}lC_v) = \eta C_v \quad (3.2)$$

From the above expression, the rate of heat loss can be calculated Buddenberg [29].

$$(i^2R)/J = 2\pi kL(t_f - t_c)/[\ln(r_c/r_f)] \quad (3.3)$$

where

- $i$  = Current in the filament
- $R$  = Resistance of the filament
- $J$  = mechanical equivalent of heat
- $t_f, r_c$  = Temperature of the filament and cylinder wall, respectively.
- $r_f, r_c$  = Radius of the filament and cylinder
- $L$  = Length of the filament.

At constant current, the resistance changes as the thermal conductivity varies. The temperature depends on the thermal conductivity of the gas and of resistance which are given by:

$$K_t = k_0(1 + \beta t) \quad (3.4)$$

and

$$R_t = R_0(1 + \alpha t) \quad (3.5)$$

where

- $0$  = Refers to some standard temperature
- $t$  = Average temperature of the gas in  $^{\circ}\text{C}$
- $\alpha$  = Constant
- $\beta$  = Constant

### (1) TCD Cell Design Modification

The Katharometer cell was surrounded by asbestos blocks. Figure 3.4 shows the asbestos blocks which accommodates the TCD outside the oven. The blocks were machined to enable an electric blanket to be placed in blanket to be placed in a position surrounding the TCD.

Another aspect which has to be taken into account is that the cell has to be completely sealed to minimise heat loss. The third asbestos block (cell cover) had holes drilled through it to enable the Resistance Temperature Detector (RTD) probes to monitor the temperature of the cell.

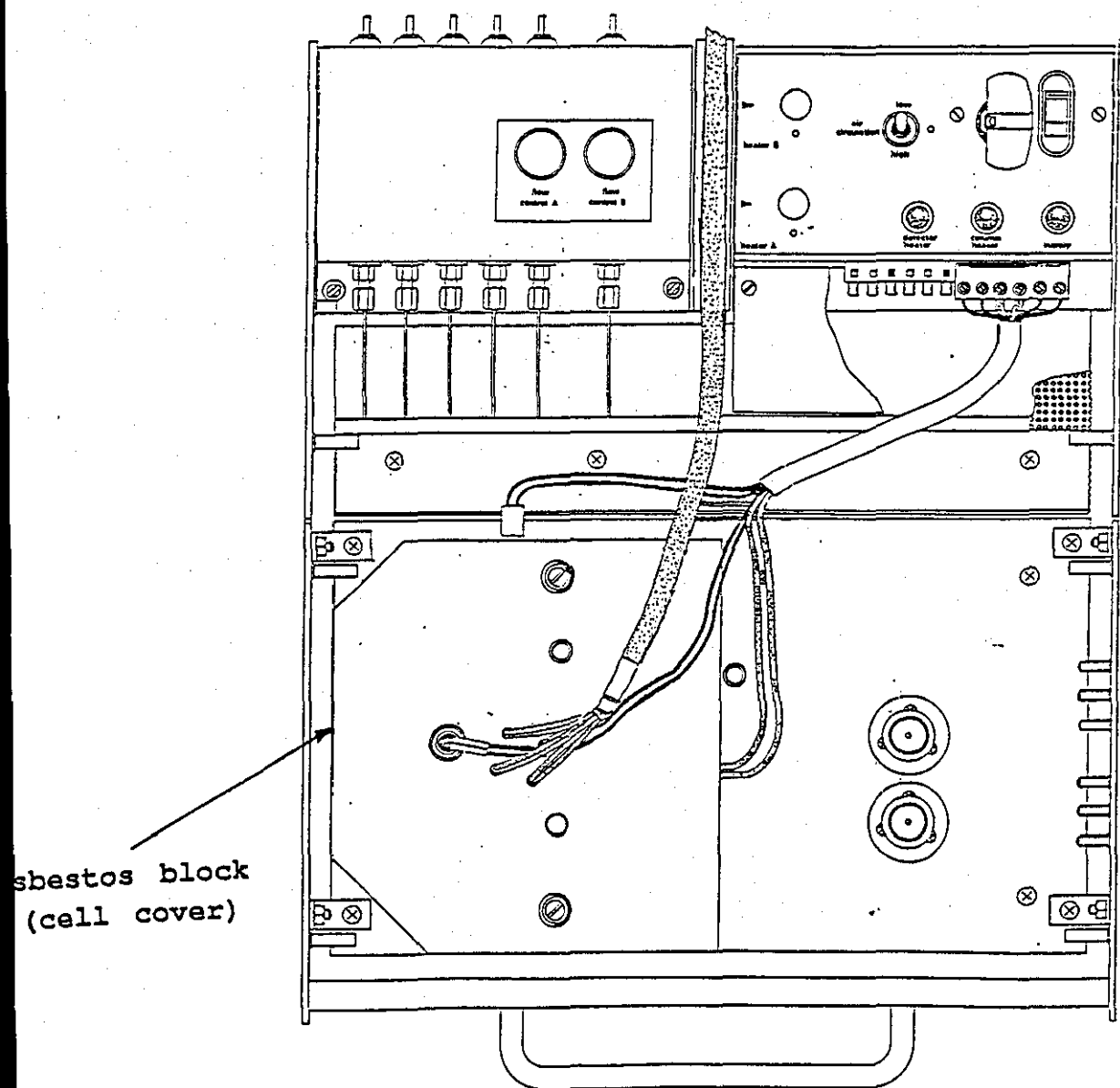


Figure 3.4 Plan View of Katharometer Cell

## (2) The TCD (Katharometer) Calibration

Measurements were carried out to find the sensitivity of a katharometer to flowrate and composition changes. It was observed that the katharometer is sensitive to composition rather than flowrate. If two streams are flowing at identical rates and then only if the unbalance exceeds about 0.5% does the katharometer show a measurable response.

## (3) Temperature Measurement

The Pye 104 Oven temperature and the Thermal Conductivity Detector Cell temperature are measured by two Resistance Temperature Detector (RTD) probes inserted into the holes provided at the top of the Oven and into the side of the Katharometer cell. With the present apparatus used for these experiments the temperatures are measured and set separately. The Oven and Katharometer were then set to the required temperatures by appropriate temperature dials. The RTD probe is physically connected to the terminal box panel (T.35).

### 3.3.3 The Valco Switching Valve

The Valco three-port rotary switching valve is employed to add the perturbation flow to the column and also to remove it from column. The valve is designed for use in a gas chromatographic applications. This device is constructed for high performance operation of temperatures up to 175° C and a pressure of 1000 psi (70 bar) with, zero dead volume.

The Valco switching valve is constructed from a high Nickel / Chrome Hastalloy C alloy which provides superior chemical resistance. During operation the device was found to be more practical than the SPECAC 6-port valve, which was used in the early stage of this project. The SPECAC valve shown in Figure 3.5 was replaced due to the leakage of gas through the seal at the base of the handle. This caused the recorder to give unrealistic readings. Using the P-type 3-Port switching valve eliminated the gas leak problem.

The Life span of the Valco switching Valve is 25,000 - 50,000 operation [12]. Under normal operations using gases

The life span of the Valco switching Valve is 25,000 - 50,000 operation [12]. Under normal operations using gases it will last much longer, without requiring any major maintenance. However, for disassembling the Valco Valve shown in Figure 3.6 a, the following procedure was followed :

- (1) Two locking nuts 1 and 2 was separated using two spanners, until the spring rotates freely.
- (2) With valve still assembled the sealing surfaces were taken apart by cycling the valve.
- (3) The valve was held as in Figure 3.6 b bracing the rotor in valve body (threads down).
- (4) The rotor was then lifted straight up and out of valve body.

When the valve body is completely disassembled, maintenance can be carried out by cleaning the rotor and valve body using compressed air, to blow out any contamination present. In reverse order the valve was reassembled, however it was necessary to test the valve after reassembly . One of the most important steps is to make sure that the valve is free of gas leaks. Gas leaks contribute to loss of gas, noise, and drifting of baseline.

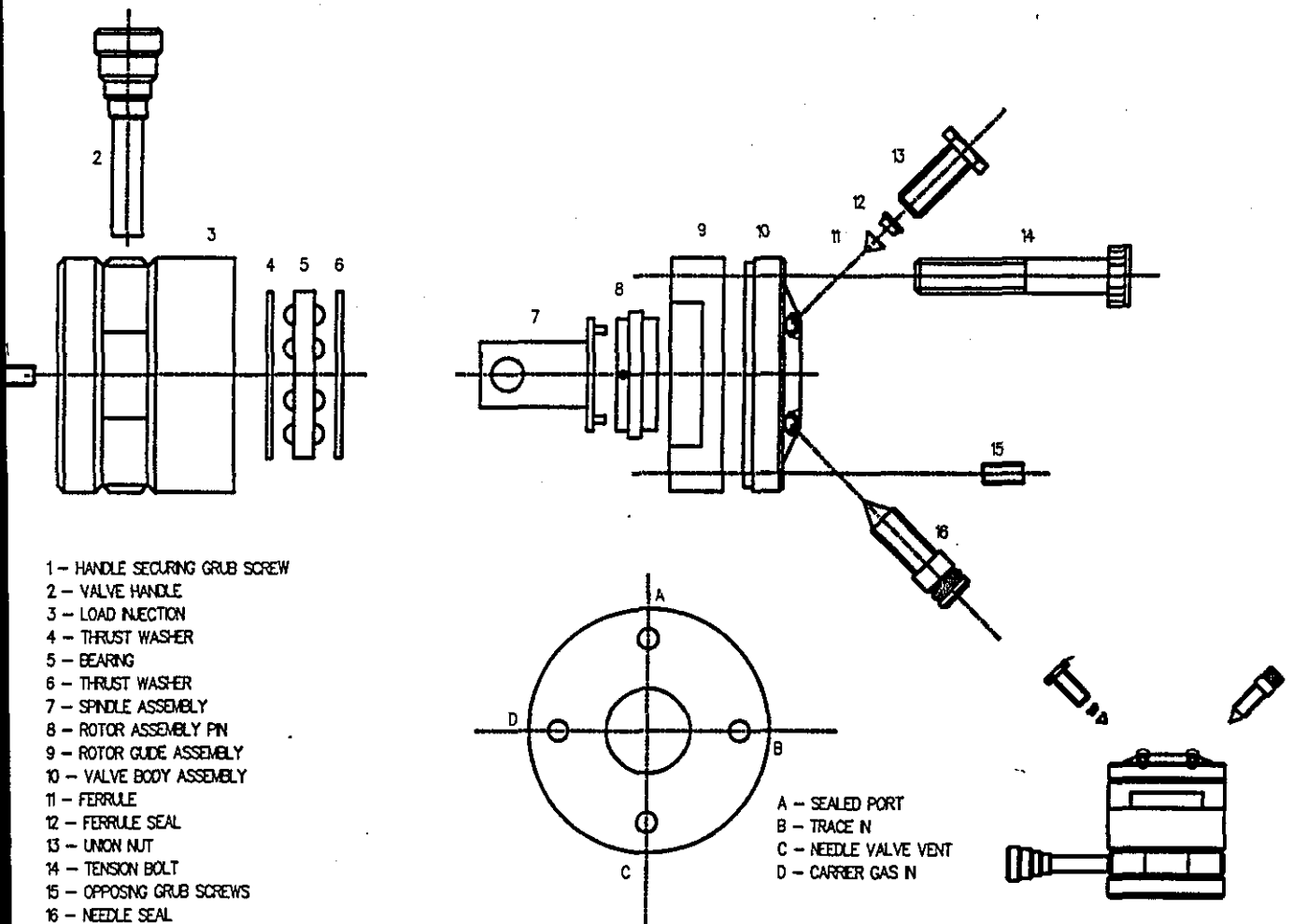


Figure 3.5 The Overall View of SPECAC Valve

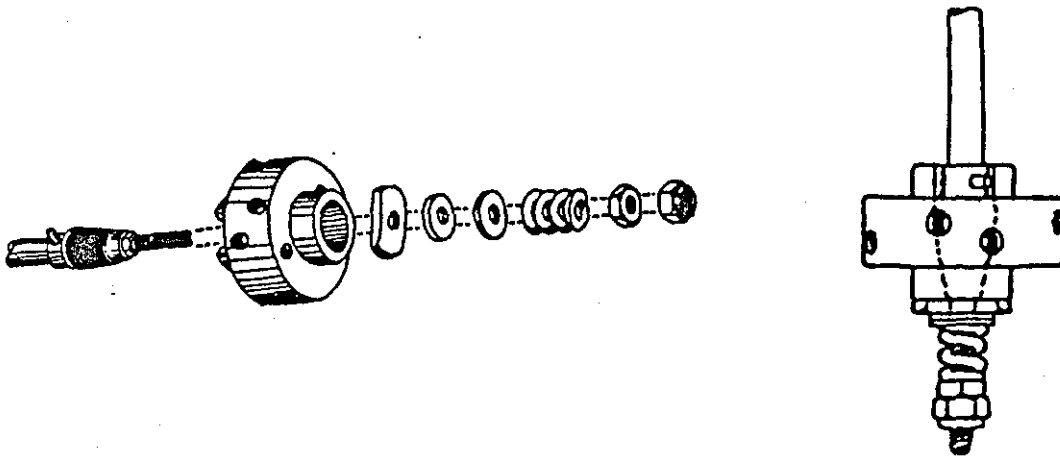


Figure 3.6.a A Valco Switching Valve

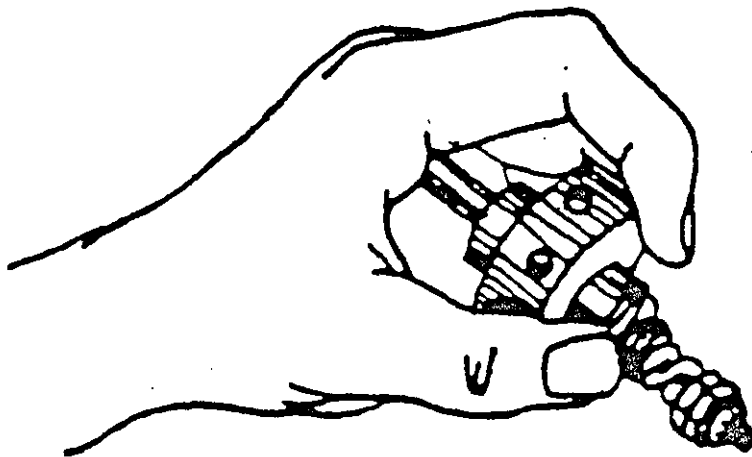


Figure 3.6.b The Valco Switching Valve Handling Procedure

### 3.3.4 Perturbation Gas Trace Mixing Device (First design)

The purpose of Trace Mixing Device (TMD) is to enable the perturbation flow to be sharply cut off when the flow is switched to the vent. Previously, there was always a "dead volume". After the perturbation gas flow was switched off some of the gas would continue to diffuse out from the dead volume.

In the experiments, the dead volume causes the response when adding the perturbation flow to differ from the response when cutting the flow off. If the responses are mirror-images of one another there the dead-volume is so small as to have no effect. It was hoped that the new trace mixing device would solve this problem. Preliminary calculation and design of the TMD was carried out as shown in Figure 3.7. Two pieces of brass; one a 1/2 inch hex brass bar of 45 mm in length and cross section of 12 mm was cut to size. The bottom end was turned down to 5 mm diameter and to 15 mm in length, with 3 mm x 60° chamfer. This bottom end also threaded to 10 UNF thread size, to fit the switching valve port, and to allow it to be screwed in as far as possible.

The brass hex bar was then further drilled to a depth of 20 mm, but then tapped to enable a Swagelok male connector to fit on to the end of the block. The thread was 1/8 inch NPT female thread. At the end of the male connector, there is 1/16 inch plastic ferrule. As the male connector is screwed in, this ferrule is forced against the inside of the next surface. Hence, there is sealing here.

The bulk flow enters through the side arm tubing. It emerge and mixes with small flow of perturbation gas which is leaving the valve port. The mixed gas then flows out through the axial tube. The side arm tube was 1/16 inch stainless steel piece which was brazed in place in order to seal it. A swagelok 1/16 inch to 1/8 inch reducing union could then be attached to this tubing, so that the bulk flow could then be connected.

Experiments made with this device did not quite give mirror image responses and showed that although the device reduced the dead volume it did not completely eliminate it. Therefore a modification to the design was made to reduce the dead volume further.

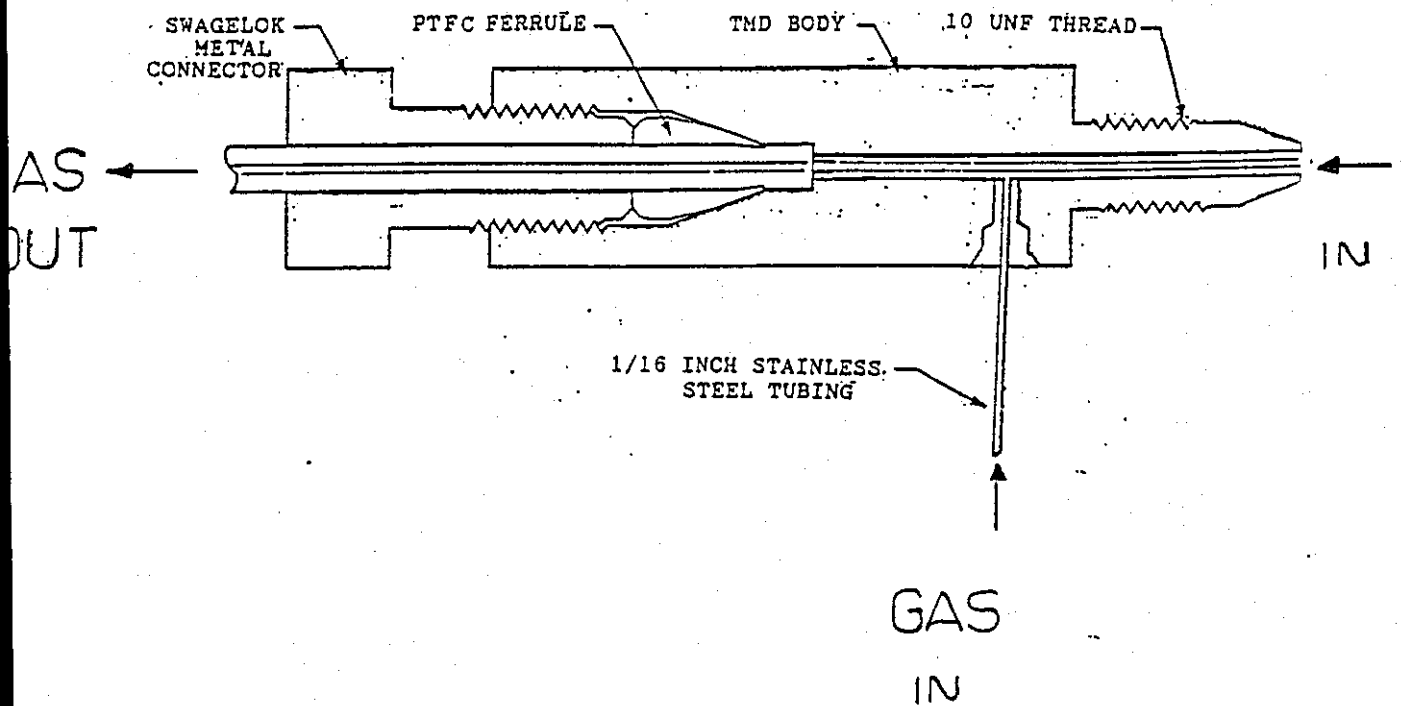


Figure 3.7 Internal Configurations of two piece perturbation gas Trace Mixing Device (TMD)



(1) Perturbation Gas Trace Mixing Device (Second design)

There were many problems concerning the first design:-

One of the main problem areas of the previous trace mixing device was the "dead volume" between the valve outlet port, from which the perturbation gas flow emerged and the inlet and outlet ports of the mixing device. Although the perturbation gas flow is cut off cleanly the effect of the dead volume makes the flow responses to addition and cut-off different. Even after the flow has been cut off, gas will effectively still be flowing due to diffusion of gas from the dead volume. On addition of perturbation gas the volume which was dead volume to cut-off becomes active volume as the perturbation gas flow sweeps through it.

Another feature which caused concern in the first design was the axial tube seal, where the two pieces joined together, gas leaks could occur. Since we are sensing very small flows. The TMD must be completely free of gas leaks.

Similarly, it was noted that when the 1/16 inch plastic ferrule which was forced against the inside of the hex outer face by the male connector it was surrounded by a large empty space. Also gas could be trapped in between the two pieces of the brass chambers. To overcome this problem the plastic ferrule and the surrounding empty space was eliminated.

Finally there was the problem of the 1/16 inch stainless steel tubing which was inserted from the centre of the Swagelok male connector to the inside of the female 1/8 inch thread chamber. Previously, this axial stainless steel tube was fixed in position and could not be adjusted.

A new mixing device was designed to overcome some of these problems. The key design change was the centre of the TMD was drilled straight through. This enabled the 1/16 inch stainless steel tubing to be inserted axially through the device, and to penetrate into the switching valve port. To do this it has to be adjustable to the desired depth.

In this arrangement the bulk gas flow enters through the side arm. It then flows down the outside of the axial tube and into the valve port. The perturbation gas flow enters at this point. The mixture then leaves through the axial tube to the outlet.

In this design the "the cut off position" takes place at the bottom of the port and as close as possible to the "mixing point" thus reducing the dead volume. Also, after the gas is mixed there is no dead volume in the system. The device will not work if flow enters through the axial tube and leaves through the side arm because of the dead volume in the annular space around the axial tube. Figure 3.8 shows the eventual successful perturbation trace mixing device (TMD) It eliminated the dead volume, and enabled all trace addition and removal runs to be mirror images of each other.

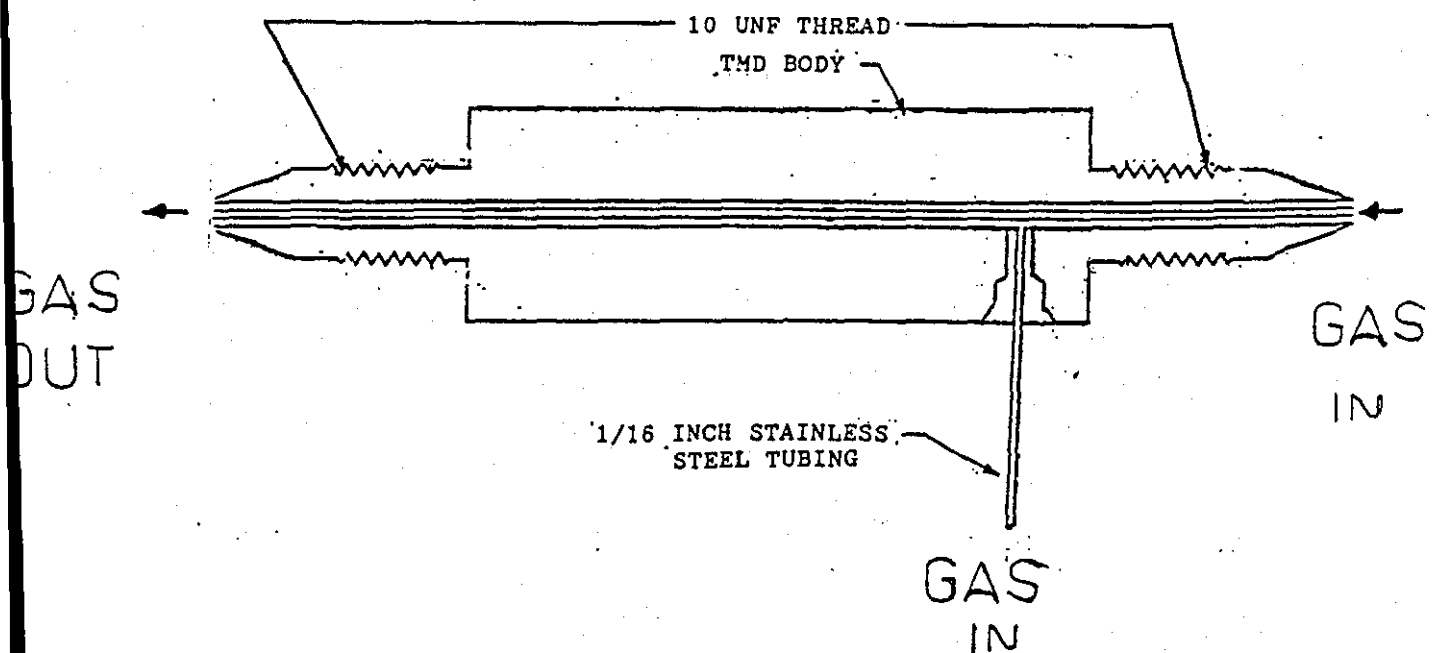


Figure 3.8 The New Trace Mixing Device (TMD)

## (2) Chemical Treatment of Trace Mixing Device

After welding the stainless steel tubing using super Silweld 1618, chemical treatment of the TMD was required to remove any excess soldering blockage in the TMD and to retain its original colour. This was achieved by placing the device in 15 - 20% Orthophosphoric Acid overnight, followed by thorough rinsing in water and drying. Finally the part was vacuumed for 20 mins.

### 3.3.5 Capillaries

#### (1) Upstream Capillaries

The ratio of the pressure drops through the upstream capillaries, column and downstream capillaries was found to be 120 : 4 : 1.

For a flow of 25 cm<sup>3</sup>/min the pressure drop across each column was obtained, both being approximately 0.04 bar. The upstream capillaries (two matched stainless steel) tubes of 45 inch length, 0.01 inch inside diameter were connected using the appropriate Swagelok fitting were added to the apparatus). In practice the chokes cannot be perfectly matched so two Vernier Fine Tuning Needle Valves, are included to provide a means of balancing the flows in each channel. The pressure drop across each needle valve was small relative to that across each choke. The needle valves which are very sensitive, were used to ensure that any discrepancies in the flowrate in either channel would be compensated for precisely.

#### (2) Downstream Capillaries

The pressure drop across the downstream capillaries was approximately 0.01 bar for 25 cm<sup>3</sup>/min flowrate. These capillaries consist of two matching pieces of stainless steel tubing, each approximately 12 inches in length and with internal diameter of 0.02 inch.

To act as an accurate differential flowmeter it was essential that the pressure drops through both capillaries were identical. The two tubes lengths were matched by measurement and two sections of tube were accurately matched for length.

### 3.3.6 Differential Pressure Transducer(DPT)

The pressure transducer used to detect changes in the flow rate Figure 3.9 was a Furness FCO 40 with a range of  $\pm 100$  mm of water. A chart recorder was connected to monitor the electrical output from the transducer.

#### 3.3.6.1 Transducer Calibration

The transducer output reading is proportional to the pressure difference across the transducer, i.e. the difference in pressure at two inputs to the transducer. Hence the reading on the chart recorder is proportional to the difference in the flowrates of the two streams at any time. The calibration was used to find out what factor converts chart-reading to volumetric flowrate difference.

Thermal error might have a major effect on accuracy. However the transducer used here, has a part of its circuitry components to compensate for thermal error. The maximum error per degree over the temperature range of its validity, was 0.1%.

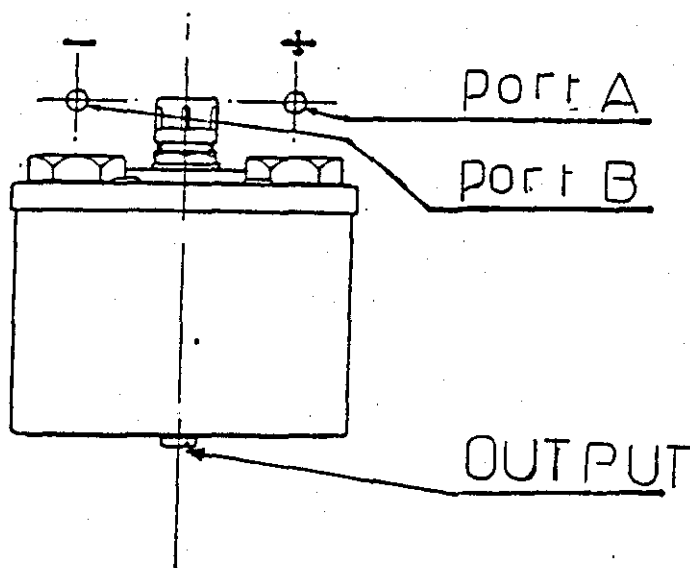


Figure 3.9 Cross section of transducer

### 3.3.7 Back Pressure Regulator (BPR) Downstream

A back pressure regulator was used to isolate the apparatus from random fluctuations in atmospheric pressure. The Porter type model 9000 shown in Figure 3.10.a is designed for low gas flow regulation and partly reduced the excessive noise levels caused by atmospheric pressure changes during experiments. However, the problem of noise produced by gas pressure fluctuation was not completely solved.

The hole in the dome of the back pressure regulator had been sealed by sticky tape to prevent any rapid fluctuations in the atmospheric pressure and noise cause by wind, opening and closing door etc, from reaching the diaphragm. But this was unreliable because the seal was only partially effective.

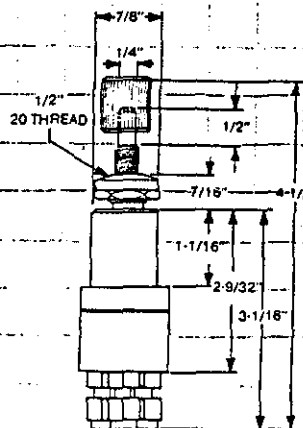


Figure 3.10.a Back Pressure Regulator (BPR) Porter Type Model 9000

Two BPRs are employed in the experiment set-up. These set the pressure at the upstream and downstream ends of the system, and so determine the molar flowrate through the system. It is easier to explain in terms of the molar flow rate because this quantity is constant every where. By the ideal gas equation :

$$Q = MRT / P \quad (3.6)$$

where  $Q$  = volumetric flow rate  
 $M$  = molar flow rate  
 $R$  = ideal gas constant  
 $T$  = absolute temperature (K)  
 $P$  = absolute pressure

The molar flow rate is constant but the volumetric flow rate will vary with pressure and temperature through the system. The Poiseuille equation relates volumetric flowrate with the predetermined upstream and downstream pressures, viscosity, temperature and the system constant. The ideal gas equation, above, relates volumetric to molar flow, hence:

$$P_u^2 - P_d^2 = KM\mu T \quad (3.7)$$

where

$P_u$  = upstream absolute pressure

$P_d$  = downstream absolute pressure

$K$  = system constant depending upon geometrical and packing constant.

$\mu$  = gas viscosity

$T$  = absolute temperature (K)

Using sticky-tape is not very efficient, and improvements were sought. One involved employing a gas tight sliding seal in the chamber.

### 3.3.7.1 The (BPR) Design Modification

The re-design and the modification to the BPR was a necessary step in reducing the effect of small pressure fluctuations on the flow rate noise level. Previously, a loose brass "hat" was placed on the top of the spring, in order for the handle to screw down on the spring. However, the chamber is open to the atmosphere by two sources.

Firstly, there is a small hole in the chamber. Secondly, the thread was not air-tight. Figure 3.9.b shows the main features of the new design, which consist of the following:-

- (1) Reference chamber
- (2) Sliding seal
- (3) Intermediate block
- (4) Countersunk brass disc
- (5) Spring
- (6) Rubber cap
- (7) Metal Disc
- (8) Four Flat Top Screws



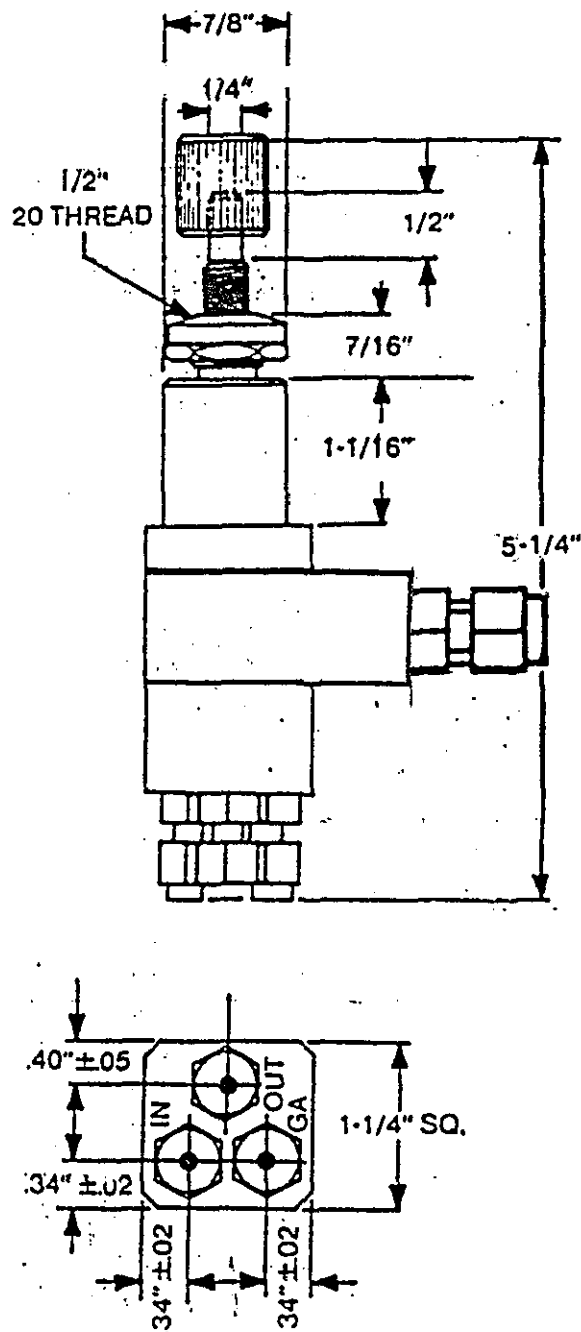


Figure 3.10.b The Re-designed Back Pressure Regulator

An intermediate block was inserted to extend the dome length of the BPR. The dome held the original spring mechanism plus a new plastic seal. The intermediate block was required to compensate for the height of the new plastic seal. The intermediate block was also drilled sideways and the side hole fitted with a sealable pipe fitting. This fitting can be plugged off or alternatively connected up and pressurised to a gas supply, thus, "dome loading" the back pressure regulator.

In an alternative design a countersunk brass disc 15 mm diameter was used on top of an air tight rubber cap which was taken from the hydraulic braking system of a car (Mini part number is M85641). This rubber seal replaced the machined plastic seal which was prone to leak occasionally.

The purpose of the brass disc is to protect the rubber cap otherwise it would be punctured by the threaded stem. Vacuum grease was used to lubricate the rubber seal and enable the rubber cap to slide easily. This arrangement was found to significantly reduce the noise due to pressure fluctuations. It did, however, suffer from hysteresis and this made the back pressure difficult to set to a particular value.

### 3.3.7.2 Back Pressure Regulator Operation

The modified Back Pressure Regulator used to isolate the system from atmosphere at the carrier gas exit. By connecting the flow-detecting capillaries together and venting them through one port of the back pressure regulator. It has three ports, inlet, gauge and outlet (see Figure 3.10.c). The diaphragm has a valve seat which blocks the outlet when there is insufficient gas pressure to force the diaphragm away from the valve orifice. The diaphragm is loaded via a spring which is loaded in turn by a screw. The strength of spring determines the maximum back pressure attainable. The reference pressure, therefore, is atmospheric pressure plus the contribution of the spring. Consequently any change in atmospheric pressure will effect the pressure at which the regulator vents.

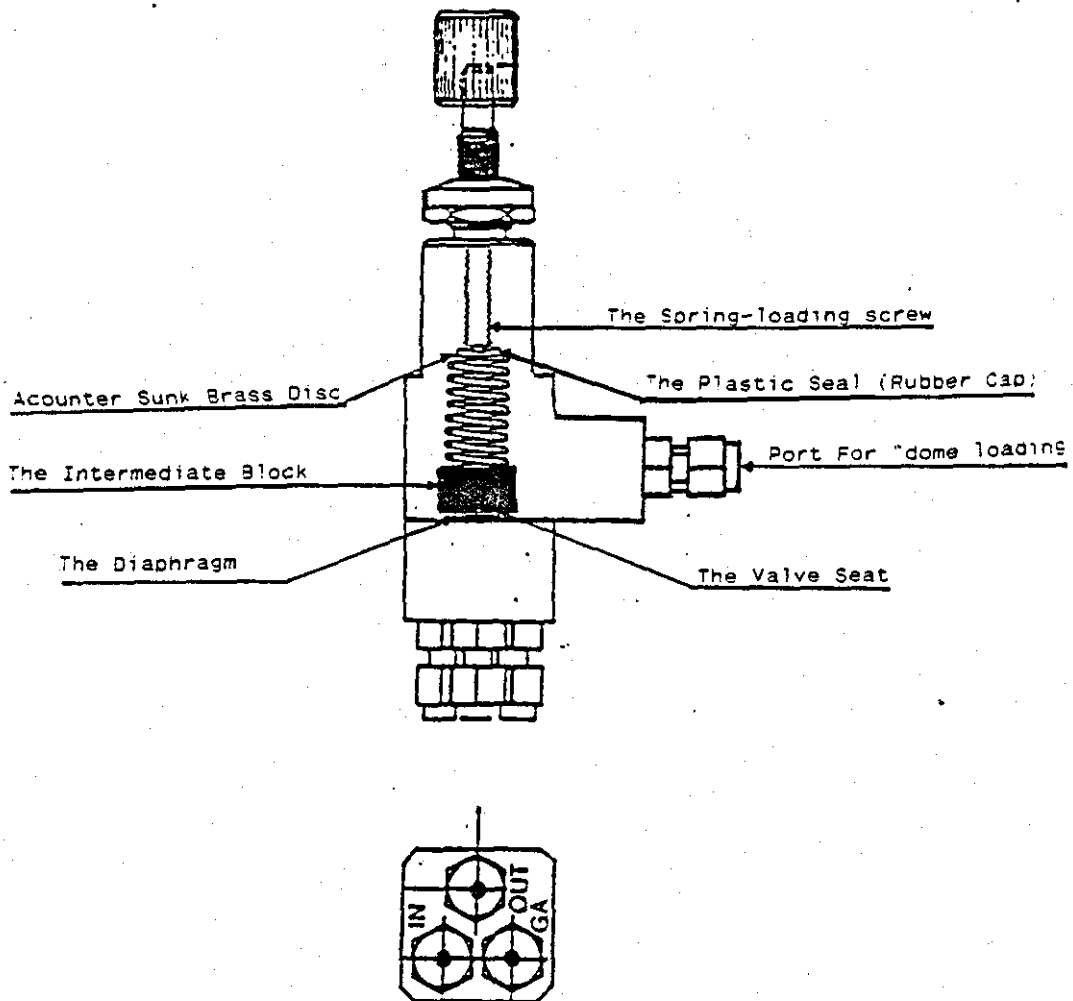


Figure 3.10.c Internal Configurations of a Back Pressure Regulator

### 3.3.8 Mass Flow Regulator (MFR)

A Condyne mass flow regulator was employed to produce the perturbation gas flowrate. This mass flow regulator has a combination of a needle valve (to set the range) as well as the differential pressure regulator (to precisely adjust the flow).

### 3.3.8.1 Mass Flow Regulator (MFR) Calibration

It is important to calibrate the MFR to determine the relationship between the molar flowrate of gases He, Ar, and the MFR setting. The system was subjected to three tests and these test were carried out with different gas flows, each inlet gas line was assigned a particular gas and the MFR was calibrated for that gas. At different MFR setting's the flowrate of each was measured with a bubble flow meter and then the volumetric flowrate was converted to a molar flowrate using the ideal gas equation giving the following graphs in Figure 3.11. The needle valve was fixed for this calibration and not subsequently altered.

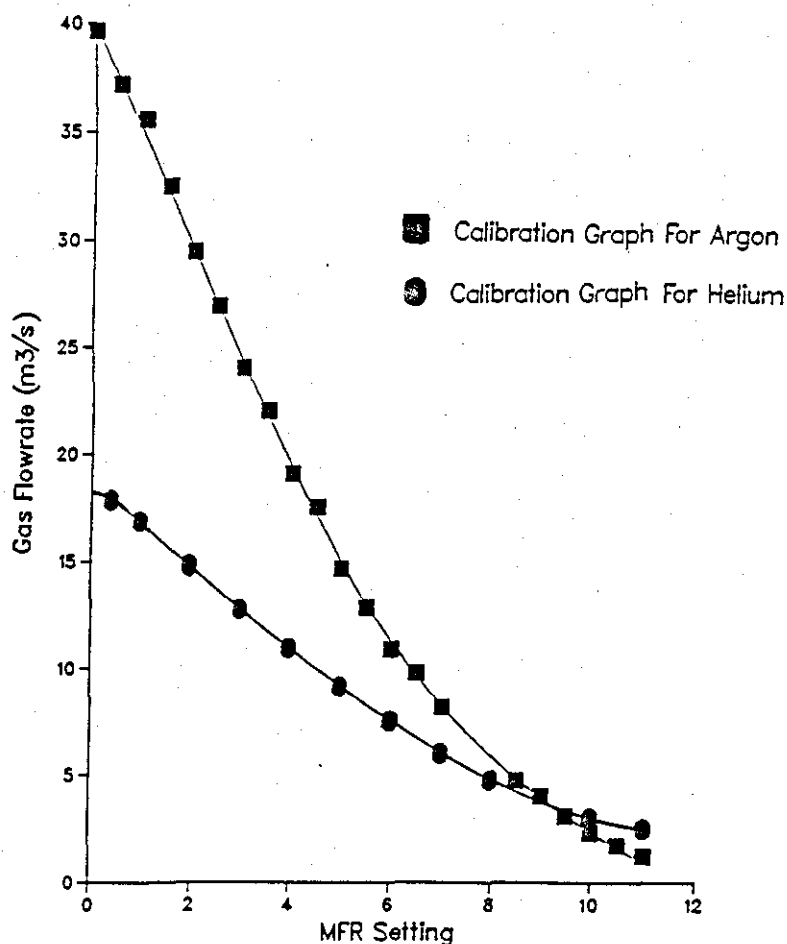


Figure 3.11 Show the (MFR) calibration using different gses

### 3.3.9 Bubble Flow Meter

Flowrate was measured with a bubble flow meter, which is a uniform glass tube containing a soap bubble. Soap solution filled the rubber teat at the bottom of the glass tube. When the teat was squeezed, a soap film was produced that rose steadily up the tube giving a visible indication of the gas flowrate. The flow rate was calculated by measuring the duration of the bubble travel between two marks at a known volume apart. Two times were measured for each flowrate in order to reduce random errors.

The inside diameter of the glass tube is about 15 mm and this will give reasonable transit times for typical carrier gas flows. However, for the small flows of perturbation gas, the bubble would take many minutes, or even hours, to travel between the two marks. To solve this problem, a small pipette can be fitted inside the glass tube. This seals on the lip at the bottom of the glass tube and so ensures that all the flow goes up through the pipette. The pipette must be centralised in order to seal at the lip, and it is kept in position by a rubber bung at the top of the glass tube.

A typical pipette had a total volume of 1 ml (with 0.01 ml subdivisions), compared with the flowmeter volume of 25 ml (with 5 ml subdivisions). The bubble flow meter gave the absolute flow rate. Any change in the flow rate in one channel relative to the flowrate in the other is detected by using the sensitive differential pressure transducer which forms part of the flow meter sited near the outlets of the column.

The columns outlets were connected to a standard Pye semi-diffusion katharometer. The outlets from the katharometer continued to delay lines, 10 m lengths of 3 mm outside diameter of Nylon tubing. The outlets of the delay line continued to two further capillary chokes. A sensitive differential pressure transducer (DPT) was used to detect changes in the pressure.

The transducer was coupled with two matched low resistance capillary tubes. A back-pressure regulator is used downstream of the flow detector to isolate the system

from atmospheric pressure fluctuations, and the entire assembly acted as a differential flow meter.

## CHAPTER FOUR

### CHROMATOGRAPHIC COLUMNS

---

#### 4.1 Introduction

The early development of packed chromatographic column began in 1958, when Golay [13] presented the theoretical basis. Since then a mass of information and experience has been accumulated in preparing high-quality columns, using different type of packing materials (refer to chapter Two). A significant improvement was made by Ettre [14]. Originally materials such as plastic, stainless steel, copper, and other metals were used for the fabrication of the outer tube of the packed column.

However, with the development of the glass drawing machine by Desty[15] glass became the material of choice. Individual investigators were then able to draw columns to the length and dimensions of choice as well as having a more inert and easily modifiable material. Although the glass columns were somewhat fragile, they essentially replaced the earlier materials and have simplified the chromatographers task for the preparation of highly efficient packed column.

#### 4.2 Columns Packings

The heart of the analytical apparatus for gas chromatography is the packing in the column, because this is where the actual separation of gas components takes place. In this research three plain glass columns of 1.5 m length, 6 mm external diameter, and 4 mm internal diameter were used, all with different modes of packing.

The particle size of the Linde 5A molecular sieve used in the two columns was about 0.6 mm and this gave a column pressure drop of about 0.1 bar at the carrier gas flowrates used. The column material packed down fairly rapidly to give a chromatographic column with an even porous structure.

To avoid the formation of air pockets within the column, the column must be packed gradually by using the following technique:

4.2.1 Column one (the reference column) with no void, was packed with 13.2 g of 5A Linde molecular sieve and had glass wool plugs (GW1 and GW2) fitted into the upstream and downstream ends, as shown in Figure 4.1 to prevent any packing from shifting. When filling, plug GW1 was fitted and the water pump vacuum tubing was attached to ARM1.

New molecular sieve was gradually poured into the up-stream end of the column ARM2. This was continued until the column was full of sieve to the required length. The column was tapped and further packing added to re-fill the column. Then a glass wool plug was inserted down ARM2 to prevent any movement of the packing.

4.2.2 Column two (the void column) was also packed with Linde 5A molecular sieve, and this column has the feature, of containing an empty void space. A glass wool plug was inserted and pushed to GW3 or GW4 either end of the column. As shown before. The void is roughly halfway down the length of the column. Water vacuum pump tubing was attached to ARM1.

New molecular sieve was gradually poured into the upstream end of the column, ARM2. This was continued until one part of the column was full of sieve. The column was tapped continuously and further packing added to re-fill the side of the column. Another glass wool plug was pushed down ARM2 to prevent any packing from shifting. Further glass wool plug was pushed down ARM1 to give a void length of about 6 cm. New molecular sieve was gradually poured into the upstream end of the column ARM1. This was continued by repeating the procedural points 2 and 3.

4.2.3 The third column was packed with glass ballotini of size 0.85 - 1 mm. In the middle there was a packed void of length 6 cm consisting of 5A Linde molecular sieve.



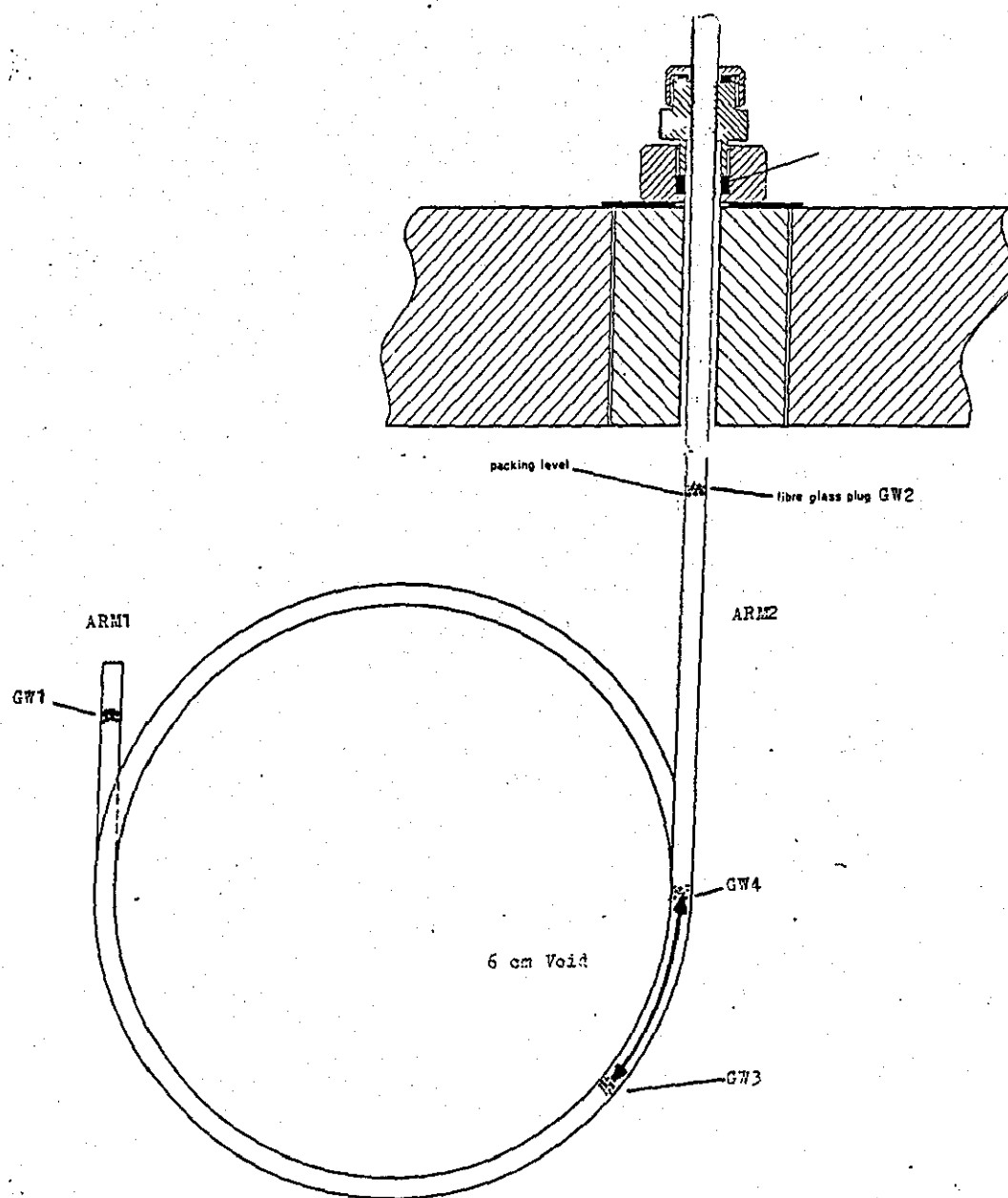


Figure 4.1 The columns packing arrangement and the packing level.

### 4.3 Chromatographic Glass Columns Mounting Arrangement

The packed glass columns were mounted as shown in Figure 4.2. The longer column end was passed through the Marinite plug and coupled to the inlet gas by tightening the hexagonal nut (finger - tight). This was a Swagelok fitting with PTFE olives (ferrules). The shorter column end was connected to the katharometer inlets by engaging the 1/16" in o.d. pipe coupling nut. All joints were checked for gas leaks using dilute soap solution before baking out the columns.

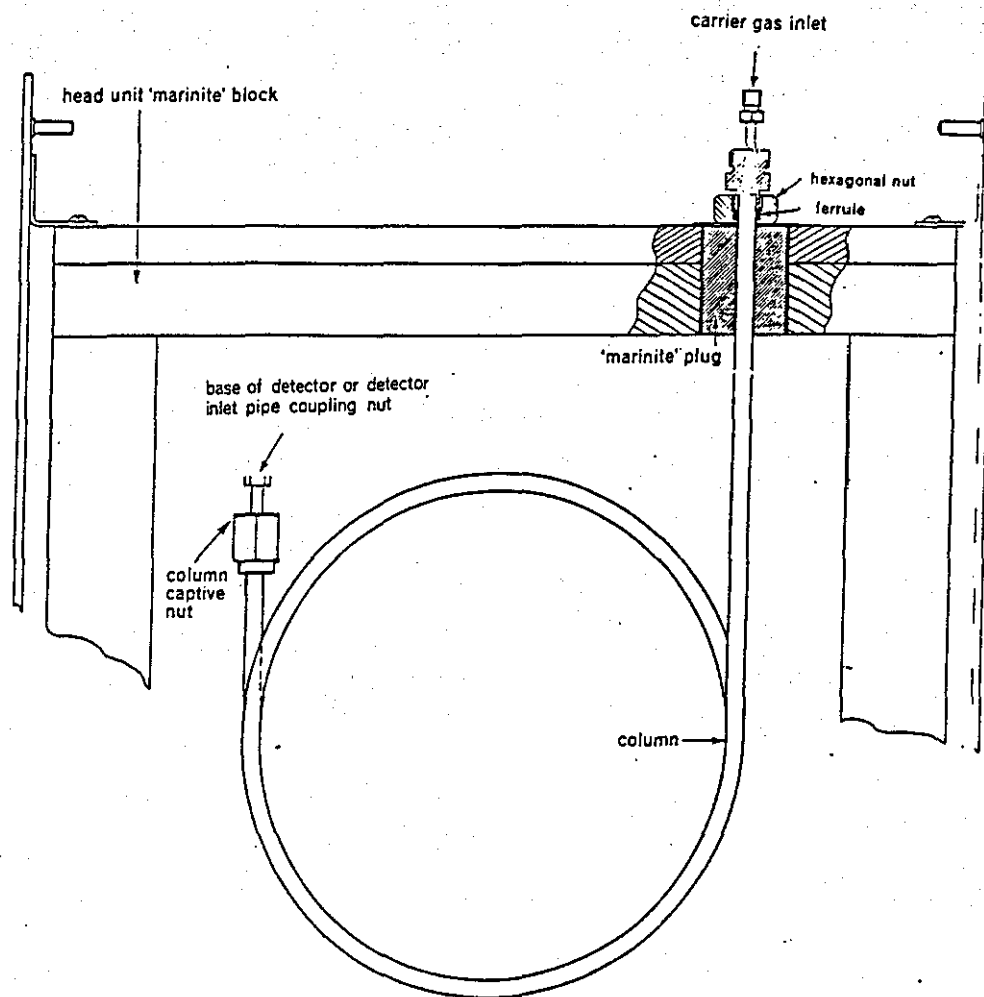


Figure 4.2 shows the method of coupling the end of the glass column by means of hexagonal nut and captive column nut.

#### 4.4 Baking Out The Chromatographic Columns

The fresh molecular sieve in the chromatographic columns was activated by heating the columns overnight at temperature of 250°C for a period of about 12 hours. There was a helium gas flow of approximately 20 cm<sup>3</sup>/min passing through each column, to ensure that no water or other contaminants remained on the packing.

The main reason for baking out the columns is to remove foreign substances from the active sites. Otherwise these sites cannot adsorb the experimental gases N<sub>2</sub>, Ar, He. This would then give incorrect readings for the residence times. Helium is the best gas for baking out water vapour because it does not adsorb on the column.

In use, column packings can pick up water from the carrier gases. When a column's packings have deteriorated to the point where they will no longer separate components, they can often be restored almost to their previous active state by placing the columns in an oven, and heating it to 250°C for a period of about 12 hours, with helium gas flowing through. In practice, it is often found to be more convenient to repack such columns with fresh material rather than attempt to regenerate the previous packing.

## CHAPTER FIVE

DETECTION OF A VOID IN A  
PACKED CHROMATOGRAPHIC COLUMN

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## 5.1 Introduction

Packed columns are used in most of the industrial and academic laboratories engaged in the separation processes of GC. Undoubtedly, the packed column is the single most important component which separates the sample species, and it remains the heart of the analytical instrument. Packed columns are also used as chemical reactors and in deep-bed filters. Usually the process performance depends on the "perfection" of a column. A perfect column is one that is uniformly porous.

To attain such quality, a column has to be prepared carefully if it is to accomplish the desired analytical results. Practical manuals are available Chapman [16] which discuss how column parameters determine efficiency. For packed columns, homogeneity of the packing is extremely important. Unfortunately, no simple method is available to check the homogeneity of a conventional analytical or preparative column. The uniformity of packed columns with transparent walls can be checked visually although this not possible for metal and other nontransparent columns.

A chromatographic technique for testing packed column by using reactive samples was proposed by Langer et al [17]. It was further developed for the detection of void zones by Patton and Langer [18]. In their method, an injected sample undergoes a first-order, thermal, gas-phase decomposition reaction to produce a product that is less strongly retained by the column than the reactant is. The product moves faster than the reactant and the chromatogram for a perfect column is a long-drawn-out, decaying response due to the product, followed by a normal peak due to the remains of the reactant. If the column has a void zone, the product travels across the gap at carrier speed, whereas in the packing it travels more slowly than

the carrier. This causes a distortion of the chromatogram that corresponds to the location of the void zone.

Recently a new technique has been exploited by Mason, Buffham and co-workers [19]. Based on flow-rate measurements, it is used for checking for imperfections in the packing of a chromatograph column, using adsorbed gases. When bands of adsorbed materials are in transit through a chromatographic column the fluctuations in the flow-rate of the gas leaving the column are monitored. With a perfect packing and constant flowrate of the gas into the column, there should be no variation in the flow rate downstream of the column while the substance is passing through the column. However if there is a gap in the column packing then the substance will be momentarily liberated resulting in an increased flow-rate downstream. The substance thereafter being readsorbed resulting in a reduced flow-rate downstream.

## 5.2 Sorption - effect Chromatography

The sorption effect in gas chromatography was first described by Bosanquet and Morgan [20] who showed that the gas velocity is greater in a region of the column where gas is adsorbed than it is in regions where there is no sorption. The sorption effect in gas chromatography recently has been exploited by Buffham et al [21]. The sorption effect is the change in local flow rate as a sample of adsorbable gas is adsorbed and desorbed in a chromatographic column. In order to measure the sorption effect, the carrier gas flow-rate and the temperatures of the columns must be kept constant.

When a sample band leaves a column, there is an increase in flow-rate caused by the desorption; both the conventional detector and the flow-meter respond together because the effluent flow-rate and composition change together. However, the sorption - effect chromatogram (the differential flow-rate record) will show additional features that do not appear in a conventional chromatogram.

This is because the flow meter responds to flow rather than to compositional transients, and so can indicate changes in the amount of sorption anywhere in the system.

Most of the molecules of a sorbable species in contact with the column packing are sorbed. Sorbed molecules occupy less volume than non-sorbed molecules and so adsorption or desorption produces a change in the volume that the sorbable molecules occupy in the column.

This causes a pause or surge in the gas flow that can be detected by the downstream differential flow meter. The measurement of such changes in flowrate is called "sorption - effect chromatography" because it depends on the sorption effect. Figure 5.1 shows a typical sorption effect chromatogram for a flow of a single sorbable species added upstream into a carrier passing through a packed column with 6 cm empty space gap.

Several changes in flowrate can be identified the first caused by adding the perturbation gas; the second is the upstream delay line (5 m); the third is when the perturbation gas enters the column and adsorption occurs; the fourth is the beginning of the void as the sorbable species leaves the packing and enters the void zone; fifth is the resorption as the band leaves the void zone and enters the packing downstream from the void; the sixth is leaving the column the seventh is the downstream delay line(10 m); and the eighth is the measuring capillaries.

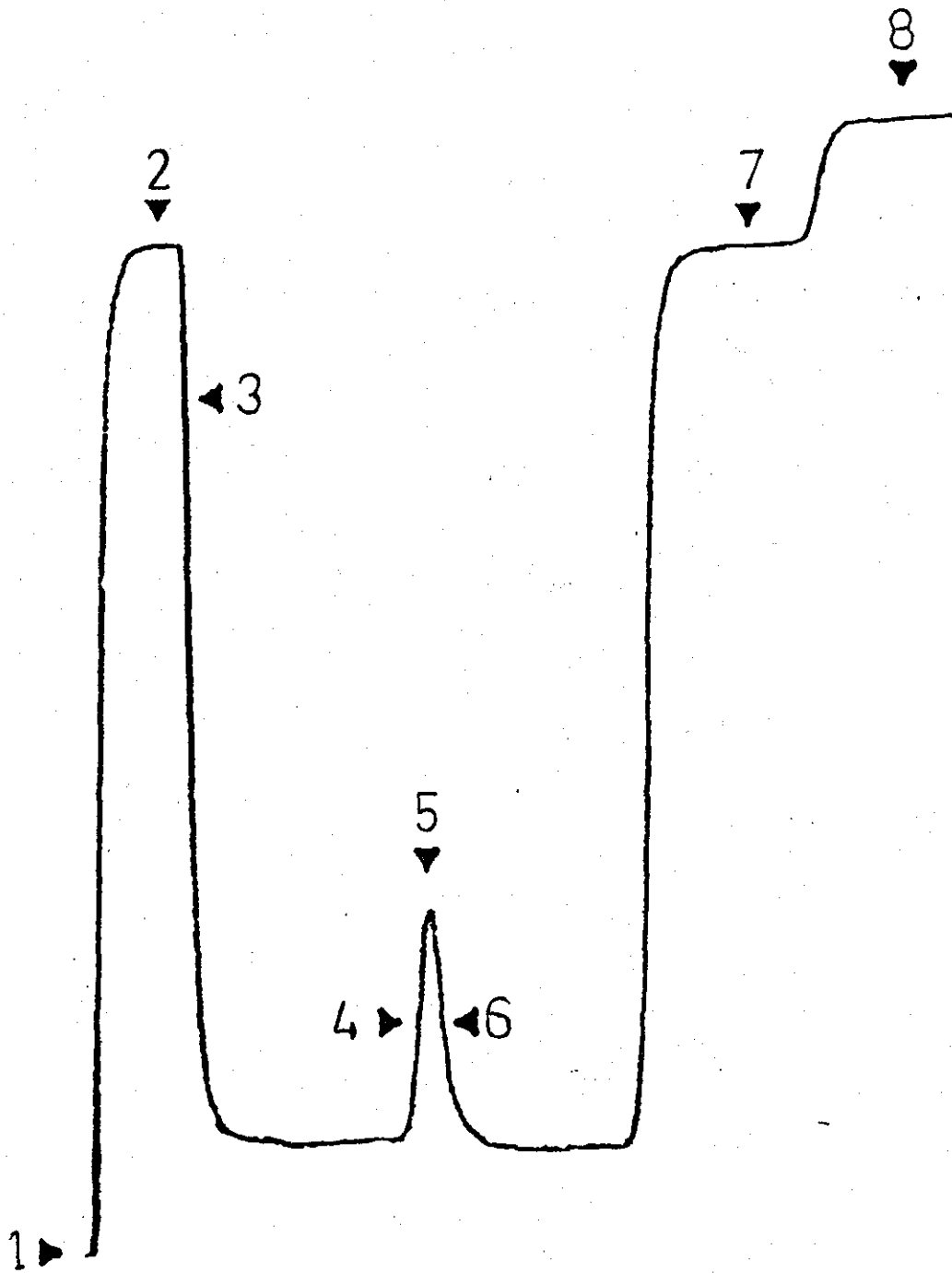


Figure 5.1 Adsorption-effect chromatogram for a flow of pure gas nitrogen added to a non-sorbed carrier helium. Responses: 1 = adding perturbation flow; 2 = upstream delay lines; 3 = sorption on entry to the column; 4 = desorption; 5 = peak caused by void zone; 6 = desorption at exit; 7 = down stream delay line; 8 = measuring capillares.

### 5.2.1 Advantages of Sorption-Effect Chromatography

Sorption-Effect Chromatography is the measurement of the total inventory changes of a chromatographic system. It is an additional chromatographic measurement and also one that is different from that provided by all conventional detectors which usually measure changes in a physical property of the outlet stream.

- (a) The sorption effect is easily observed in gas chromatography. Differential flow meters can reveal the effect which gives a response that is proportional to the molar quantity of a component as it leaves the column. The response is instantaneous and may be observed at a location remote from the end of the column.
- (b) A sample can be detected on arrival at the column and again when it leaves the column.
- (c) The sorption-effect detector is simple, rugged, and corrosion free.
- (d) The sorption-effect detectors are very small in size and reasonably inexpensive.

### 5.2.2 Disadvantages of Sorption-Effect Chromatography

Experiments performed so far with a differential capillary flow meter have revealed the following disadvantages of the method.

- (a) The method lacks sensitivity when hydrogen or helium are used as carrier gases, compared to using a katharometer detector.
- (b) The apparatus described here is very sensitive to sudden small changes in atmospheric pressure, such as those caused by opening and closing doors.
- (c) Noise caused by atmospheric pressure variations that result from an unbalance in the volumes downstream of



each column. Our apparatus is set with two matched streams. Any unbalanced drift in the carrier-gas flow-rates will cause a drift on the baseline.

### 5.3 Void Detection and Location

Figure 5.2 shows schematically a packed column with a void. When a band of adsorbable species leaves the packing and enters the void zone desorption occurs, followed by resorption as the band leaves the void zone and enters the packing downstream from the void.

The position of the void zone can found from the time at which the flowrate changes by desorption and resorption occur relative to the retention time of the flow in the column. Figure 5.3 shows a sorption-effect chromatogram for a single sorbable component added to non-sorbed carrier gas passing through a column with a void zone. Desorption and resorption have combined to give a peak on the chromatogram.



Figure 5.2 A packed chromatographic column with a void zone

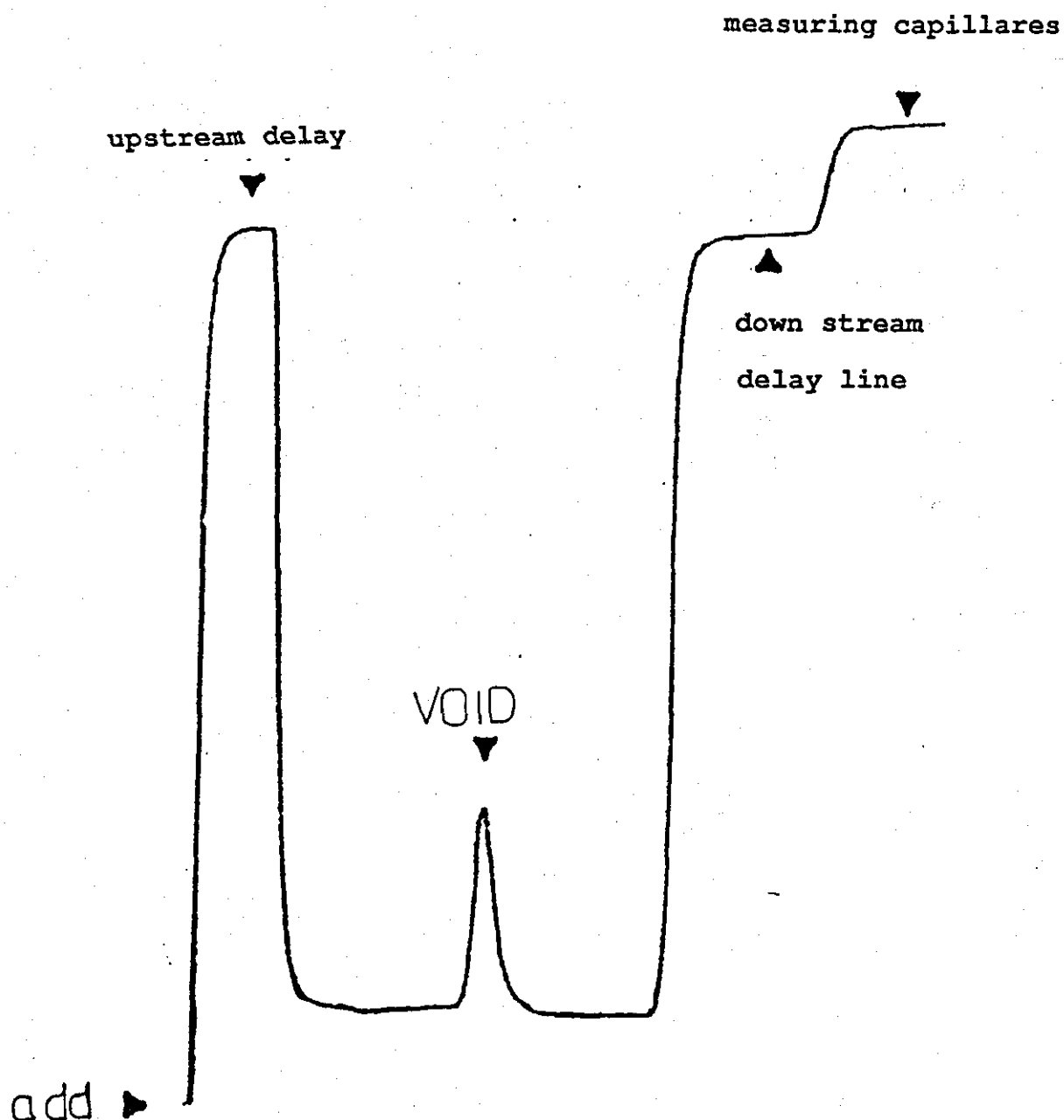


Figure 5.3 Adsorption effect chromatogram for a single sorbed front passing through a column with a void zone. The labels indicate the position of the front and the cause of the response.

Times are measured from the sorption peak caused by the sorption when the flow enters the column. Typical behaviour is demonstrated by using helium carrier gas and nitrogen as the materials adsorbed onto 5A molecular sieve at 60°C. The technique enables the void zone position in the column to be found (see Figure 5.3).

#### 5.4 Approximation of Void-Zone Volume

In sorption - effect chromatography the changes in the inventory within the column are detected rather than changes in effluent composition. It is different from conventional chromatography because the flow-meter response in sorption - effect chromatography measures a flow transient and not a composition transient.

Changes in amount of sorption are seen almost immediately they occur whereas composition changes are not seen until they reach the detector. In the present application, our main concern is to calculate the volume of a void zone. When a sample of gas is added into a desorption effect chromatograph, it is separated into its components in the usual way.

The flow-detector response is, however, rather different from that which would be given by a katharometer. As soon as the perturbation flow is added, gas is displaced and the flow detector responds. It responds again as the perturbation gas is absorbed by the column. While the sample travels through the column there is no response. The system then responds again as the gas is desorbed and adsorbed at the void in the column. This final part of the sorption-effect chromatogram shows the perturbation gas being desorbed and resembles a conventional chromatogram.

However, the integral of incremental flowrate with respect to time on sorption - effect chromatograms (i.e. areas) represent gas volumes. The simplest case is when the carrier is not absorbed, but the perturbation species is strongly adsorbed. Figure 5.4 shows the type of chromatogram to be expected when a flow of a single sorbable component is added into carrier flowing into a column that has a void zone.

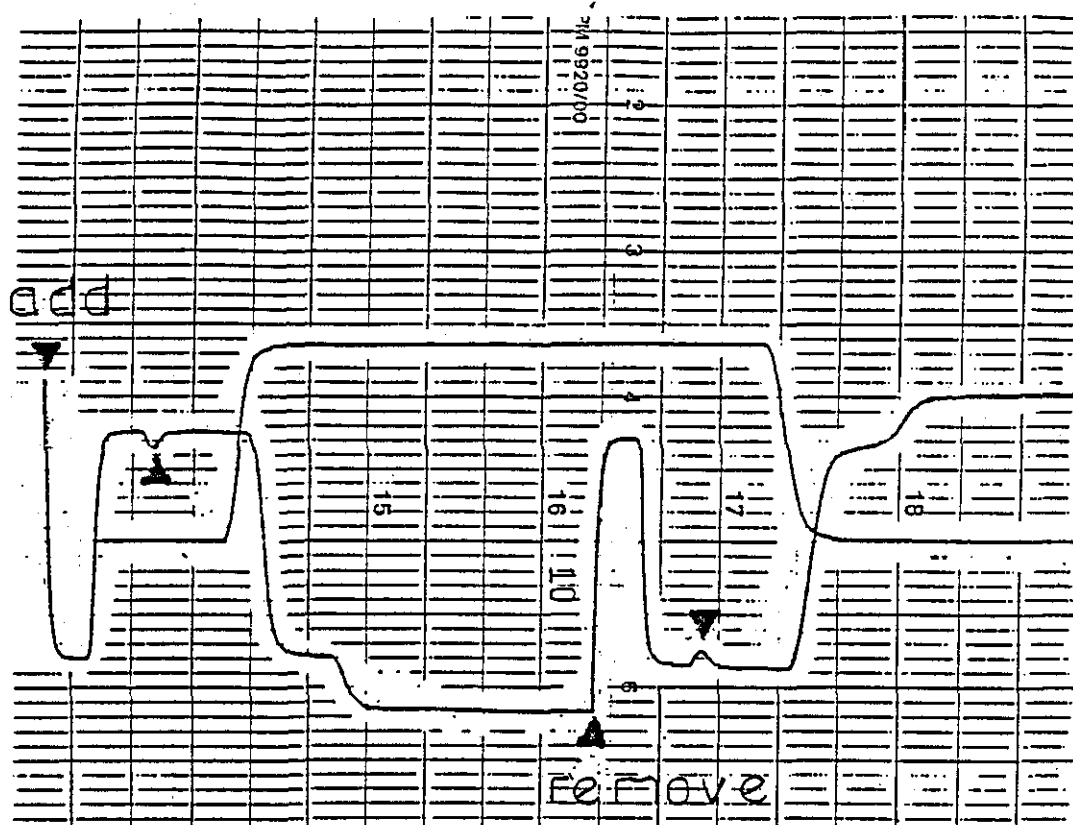


Figure 5.4 Two sorption effect chromatogram produced by a column containing a void. One is for adding the small flow of strongly adsorbed nitrogen and the other for removing the nitrogen. The carrier gas was non-adsorbed helium. Note that the two responses given by the void are (mirror-images) of one another. Such behaviour is only shown when there is minimal dispersion caused by the mixing system through which the nitrogen flows. The katharometer trace is also shown on this Figure.

## 5.5 The Use of Perturbation Band/Slug Material

So far, all of the discussion and results have been concerned with adding a perturbation flow to the system. It is now necessary to consider adding a band (slug) of material to the system.

The disturbance to the flowrate looks rather like a distorted single cycle of a sine wave. There is a small desorption peak followed by a small adsorption trough. The flow-rate response is very rapid relative to the peak widths in the system; the absorption peak occurs at the moment the sample passes the void zone and the desorption peak starts when the sample enters the void. The time interval between the time when the perturbation flow is added and the appearance of a peak on the sorption effect chromatogram depends on the position of the void zone. Provided that the carrier gas flowrate is constant, the times between responses caused by a given solute band on a sorption - effect chromatogram are proportional to the distances between the features of the column that caused the responses. The position of a void zone, as a fractional distance  $x/L$  from the column entrance, is the ratio of the time  $T_x$  for the peak caused by the void zone to appear after desorption to the time  $T_L$  required for the flow to travel the entire column.

$$x/L = T_x / T_L \quad (1)$$

The carrier and sample gas must be chosen to give a smooth baseline and a distinct peak. In sorption-effect chromatography the sensitive differential flow-meter measures changes in the gas inventories in the column. This means that thermal noise in the oven will give a noisy baseline if the carrier is adsorbed to any extent. The sample, however, must be strongly enough adsorbed for there to be strong sorption and desorption effect as the sample band enters the column, passes the void zone and leaves the column, but not so strongly adsorbed that the band moves so slowly and becomes dispersed.

There is a limit to the size of the sorption - desorption effects: it occurs when the bulk of the sample is

in the sorbed state as the band travels through the column. Strongly sorbed bands travel slowly and become dispersed because they are subject to mixing effects for a long time. In order to obtain reasonably large sharp sorption effects on a smooth baseline, it is necessary to choose a non-sorbed carrier and a modestly sorbed sample species.

## 5.6 The theories of scanning a void in chromatographic column

The passage of a composition front through a large void in a properly packed chromatographic column is represented in Figure 5.5.a. Typically a band of sorbed material, nitrogen, is driven by carrier gas, helium. The downstream flowrate fluctuates as the test band passes through the column.

The following theory has been developed by Mason and Buffham [22]. Let the deviation in molar flow-rate of the effluent as the band passes the void be  $w(t)$  and as it leaves the column be  $n(t)$ . Providing that the void position is such that  $w(t)$  and  $n(t)$  do not overlap, the response  $w(t)$  is made up of two parts. When the test band passes the upstream face of the void the inventory(moles) of sorbed material on the packing decreases, increasing the effluent flow-rate, Figure (5.5 b). When the test band reaches the downstream face the material is re-adsorbed, giving a decrease in the effluent flow-rate.

Let  $u(t)$  be the molar flow rate deviation caused by desorption and  $v(t)$  be the molar flow deviation caused by resorption. Then  $w(t)$  will be the sum of the two responses.

$$w(t) = u(t) + v(t) \quad (5.1)$$

The loss in inventory ( $N^\circ$  of moles) of the column as the band is desorbed will be equal to the gain in inventory as the band is resorbed and assuming no mixing within the void,  $v(t)$  will be the mirror image of  $u(t)$ , displaced by the transit time of the band through the void,  $T$ .

$$\text{Hence } v(t) = -u(t-T) \quad (5.2)$$

Combining 1 and 2 gives

$$w(t) = u(t) - u(t-T)$$

5.3

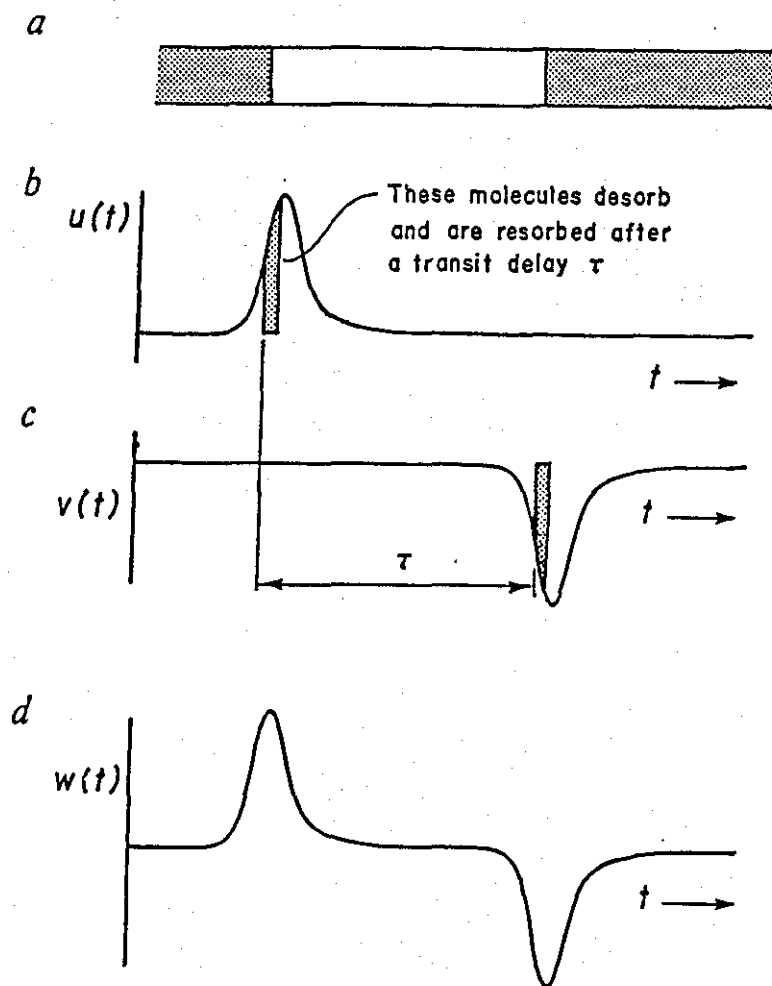


Figure 5.5 A absorbable band passing a void zone: (a) the void zone; (b) the effluent flow-rate deviation as the band desorbs into the void zone; (c) the effluent flow-rate deviation as the band is resorbed after a transit delay  $\tau$ ; (d) the total effect caused by the void zone on the effluent flowrate.

If the void zone is long, hence the time displacement  $T$  is long enough for the two contributions to occur separately, then the peak  $u(t)$  and  $v(t)$  can be seen separately. If the void is short  $T$  is smaller, and the two contributions can overlap and produce a single response. If  $T = 0$  i.e. no void then, as expected, there is no fluctuation (see Figure 5.6).

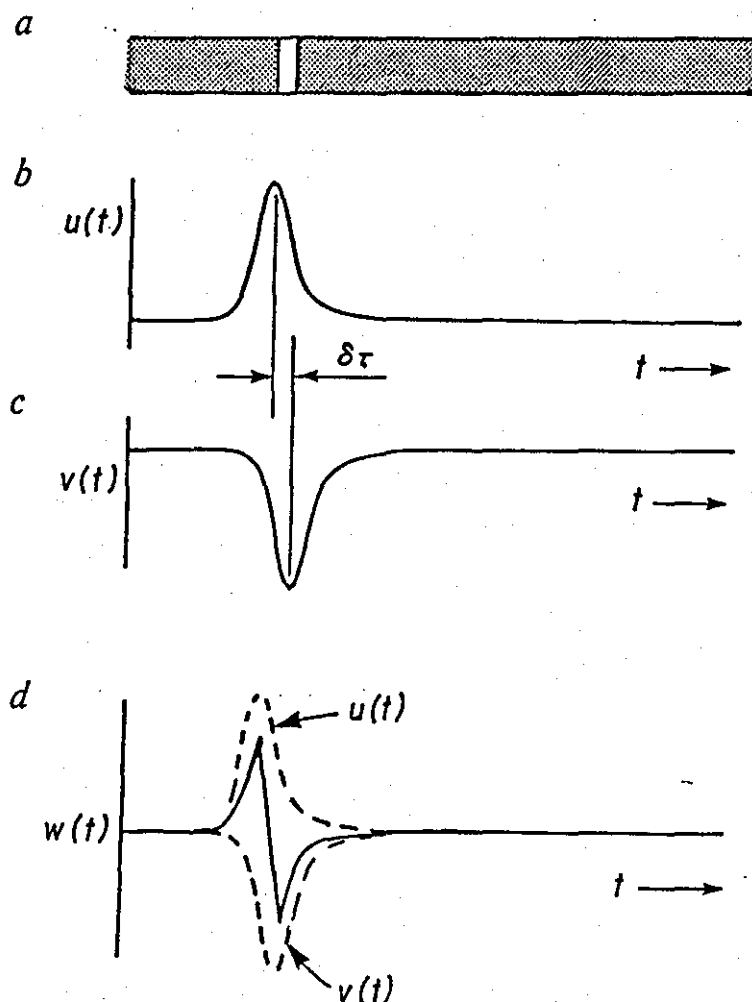


Figure 5.6 A absorbable band passing a small void zone; (a) the desorption effect; (b) and resorption effect; (c) combine to give a doublet on the absorption effect-chromatogram as shown in (d).



Let the transit time for a small finite void be  $\delta r$ . Then equation 3 becomes;

$$w(t) = \frac{d}{dt}[u(t)]\delta r \quad (5.4)$$

Integrating from time 0 to t, using  $t'$  as dummy variable, then

$$\int_0^t w(t')dt' = \delta r \int_0^t \frac{d}{dt'}[u(t')]dt' = \delta r \int_0^t du = \delta r[u(t) - u(0)] \quad (5.5)$$

At  $t = 0$  there is no flowrate deviation, and so  $u(0)=0$ .

$$W(t) = \int_0^t w(t')dt' = \delta r u(t) \quad (5.6)$$

Let this integration be  $w(t)$ , see Figure 5.7. So, by integration of the whole of  $w(t)$  the entire area under the peak may be obtained, and hence  $W(t)$ . This integration will be shaped like the peak  $u(t)$ . A second integration over the peak  $W(t)$  gives:

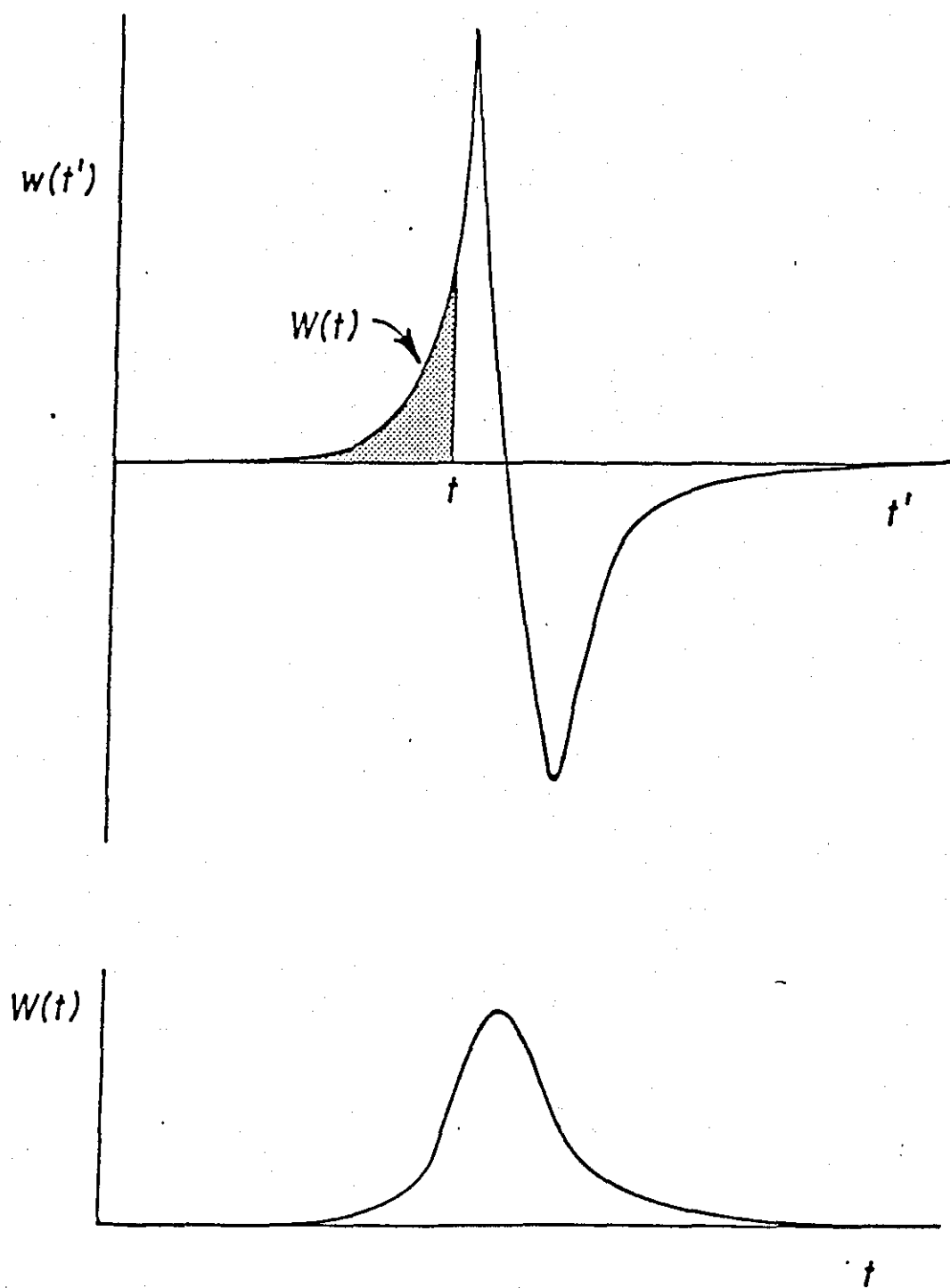


Figure 5.7 Integrating the doublet  $w(t)$  to find  $W(t)$

$$\int_0^{\infty} W(t)dt = \delta r \int_0^{\infty} u(t)dt \quad (5.7)$$

As  $u(t)$  represents the deviation in downstream flowrate caused by the change in volume due to desorption at the void, then the integral of  $u(t)$  over  $0 < t < \infty$  represents the total molar quantity in the band that desorbs. Let this molar quantity be  $N_{\text{desorb}}$ . The same molar quantity must later desorb at the end of the column, giving response  $n(t)$ . Hence,

$$\int_0^{\infty} u(t)dt = N_{\text{desorb}} = \int_0^{\infty} n(t)dt \quad (5.8)$$

Substitution of 5.8 into 5.9 gives the transit time across the void,  $\delta r$ .

$$\delta r = \frac{\int_0^{\infty} W(t)dt}{\int_0^{\infty} n(t)dt} \quad (5.9)$$

This transit time is also the ratio of the molar volume of the void,  $N_{\text{void}}$ , to the molar flow-rate,  $M$ . Provided the concentration of the sample in the test band is low,  $M$  will be constant.

$$\delta r = N_{\text{void}}/M \quad (5.10)$$

Using 5.9, 5.10 gives

$$N_{\text{void}} = \frac{\int_0^{\infty} w(t)dt M}{\int_0^{\infty} n(t)dt} \quad (5.11)$$

and (5.7) gives

$$N_{\text{void}} = \frac{M \int_0^{\infty} \int_0^t w(t') dt' dt}{\int_0^{\infty} n(t) dt} \quad (5.12)$$

The molar volume of the void can therefore be found by integrating peak  $w(t)$ , to give  $W(t)$ , and then integrating  $W(t)$  and  $n(t)$ .

In practice using a real flowmeter, the deviations  $w(t)$  and  $u(t)$  are parts of the same chromatograms measured by a single flowmeter. The calibration constant of the flowmeter,  $K_m$ , will cancel upon substitution of

$$\text{flowmeter} = K_m \cdot \text{reading} \quad (5.13)$$

If the effluent gas flow is measured at a fixed temperature and low enough pressure for the gas to be considered ideal, then the molar flow-rate is proportional to the outlet flow,  $Q_{\text{out}}$ .

So

$$M = \frac{P_{\text{out}} Q_{\text{out}}}{RT} T_{\text{out}} \quad (5.14)$$

and the molar capacity of the void zone is related to the void volume by

$$N_{\text{void}} = \frac{P_{\text{void}} V_{\text{void}}}{Z_{\text{void}} RT} \quad (5.15)$$

giving on substitution into 5.13.

$$V_{\text{void}} = \frac{Z_{\text{void}} P_{\text{out}} Q_{\text{out}} \int_0^{\infty} \int_0^t w(t') dt' dt}{P_{\text{void}} \int_0^{\infty} n(t) dt} \quad (5.16)$$

and assuming the void temperature and outlet temperature are equal, we get

$$V_{\text{void}} = \frac{Z_{\text{void}} \left[ \frac{P_{\text{out}}}{P_{\text{void}}} \right] Q_{\text{out}} \int_0^{\infty} \int_0^t w(t') dt' dt}{\int_0^{\infty} n(t) dt} \quad (5.17)$$

## CHAPTER SIX

# THE EFFECTS OF PRESSURE DROP

---

### 6.1 Introduction

It has been found that the major sources of pressure drop across the system are: the packed chromatographic column, the upstream flow setting capillary chokes; the delay lines; the flowmeter capillaries; and across the inlet of the thermo-conductivity detector (TCD).

The principle of pressure drop across the system in gas chromatography is straight-forward. When a pulse of perturbation flow is added to the carrier gas, this pulse causes pressure changes in the chromatograph. It also changes the flow rate in the chromatograph and alters the viscosity.

### 6.2 Pressure Drop Across The Column

The variation of pressure along the column, has been rigorously discussed and analysed by several authors [23]. It involves the sorption effect, and viscosity effect in a chromatographic system. The flowrate at the column inlet is constant for the duration of the experiment. However, the pressure drop across the column changes when a perturbation gas flow one of the pure components is either added to or removed from the system. The perturbation gas flow changes the composition in the column and increases the total flow rate leaving the column. The main change in composition of the material in the column arises from the addition of the perturbation flow. However, there is a secondary effect, the extra flow increases the mean pressure drop down the column and this causes an increase in the amount adsorbed. There is a further effect which is produced by the change in viscosity as the composition of the gas changes. Adding the perturbation flow to the carrier gas also change the viscosity and this may increase or decrease the mean pressure in the column.

### 6.3 Pressure Drops Across The Flow-Setting Chokes

A large proportion of the total pressure drop across the system, consists of the pressure drop across the flow-setting chokes. The volumetric gas flowrate will hardly change while the perturbation gas flow remains within the column. When gas leaves the column it passes directly into a delay line and then on to the flow-measuring part of the system. This part of the system measures the system flowrate difference, by monitoring the difference in the pressure drop across the chokes. The pressure drop across the chokes is a function of viscosity as well as flow rate. This combination of flow and viscosity is indicated by the capillary flowmeter.

### 6.4 Viscosity Effects on the Measurement of Flow Rate

An investigation of the viscosity effects on measurement of flow rate has been carried out by Mason, Buffham, and Co-worker [24]. In the experiment described (in Chapter 3) a differential flow detector was used to determine the flowrate retention time. The detector consisted of two matched capillary tubes and a pressure transducer. Such a device is sensitive to small viscosity changes as well as the small flowrate changes.

The experimental results show that when the column produces a sharp front then the viscosity effect is easier to correct for than if broader fronts are produced.

### 6.5 Viscosity Effect Across Chromatographic system

The viscosity of every gas is different hence gas mixture viscosity will depend on the composition. Because a chromatographic system contains mixtures of different composition then there will also be mixtures with different viscosity.

In addition, there is the more complex problem of the viscosity change due to the difference in gas mixture

composition caused by the composition front passing through the packed column. This causes a change in the column pressure drop. The total flowrate through the system downstream of the flow setting capillary tubes will be increased by adding the perturbation gas. Any difference in gas mixture viscosity will further change the pressure distribution.

The secondary viscosity effect is small but is significant enough to be detected by the flowrate detector, which is very sensitive to small viscosity changes as well as to small flowrate changes. At the flowmeter small changes in flow rate are detected immediately but, small viscosity changes are not detected until the composition front has reached the flowmeter. This means that the effect of viscosity on the measurement of flow rate can be greatly reduced by incorporating a delay line (a suitable length of empty tube) between the end of the column and the flowmeter.

In the past, there have been many attempts to study the effect of viscosity changes in gas chromatography. Haarhoff and van der Linde [25] have shown that the difference between the viscosities of the carrier gas and the added substances, have a substantial effect on the pressures in a packed column. The pressure across the column may be greater or smaller than that in the pure carrier gas. However, the effect on retention time is negligible.

The viscosity of gas mixtures of helium, nitrogen and argon were reported by Tohloukian, Saxena and Hestermans [26]. This extensive work has been revised by Mason, Buffham and co-workers [27]. Further study by Gibbard and Dye [28] has shown a correlation between experimental data and the theoretical prediction of Sutherland, Buddenberg-Wilke, and Chapman-Enskog [29]. It was found that the accuracy of each prediction depends on the mixture being studied. see Fig 6.1 to Fig 6.3. No theory has an overall advantage over the other.



The viscosity of gas mixture can be calculated by Wilke's and Buddenburg method of approximation:-

$$\mu_m^o = \frac{\mu_1^o}{1 + \frac{x_2}{x_1} \phi_{1,2}} + \frac{\mu_2^o}{1 + \frac{x_1}{x_2} \phi_{2,1}} \quad (6.1)$$

$$\phi_{1,2} = \frac{[1 + (\frac{\mu_1^o}{\mu_2^o})^{0.5} (\frac{M_2}{M_1})^{0.25}]^2}{2\sqrt{2} [1 + \frac{M_1}{M_2}]^{0.5}} \quad (6.2)$$

$$\phi_{2,1} = \frac{[1 + (\frac{\mu_2^o}{\mu_1^o})^{0.5} (\frac{M_1}{M_2})^{0.25}]^2}{2\sqrt{2} [1 + \frac{M_2}{M_1}]^{0.5}} \quad (6.3)$$

Where

$\mu_i$  - viscosity of components

$\mu_m$  - viscosity of mixture

$\Phi$  - correction factor

$M_i$  - molecular weights of components

The viscosities of the three binary systems are plotted along with the rate of changes of viscosity with mole fraction in Figure 6.1 to 6.3.

$$\frac{d\mu_m^o}{dx} = \frac{\mu_1^o \phi_{12}}{x_1^2} (1 + \phi_{12} \frac{x_2}{x_1})^{-2} - \frac{\mu_2^o \phi_{21}}{x_1^2} (1 + \frac{x_1}{x_2} \phi_{21})^{-2} \quad (6.4)$$

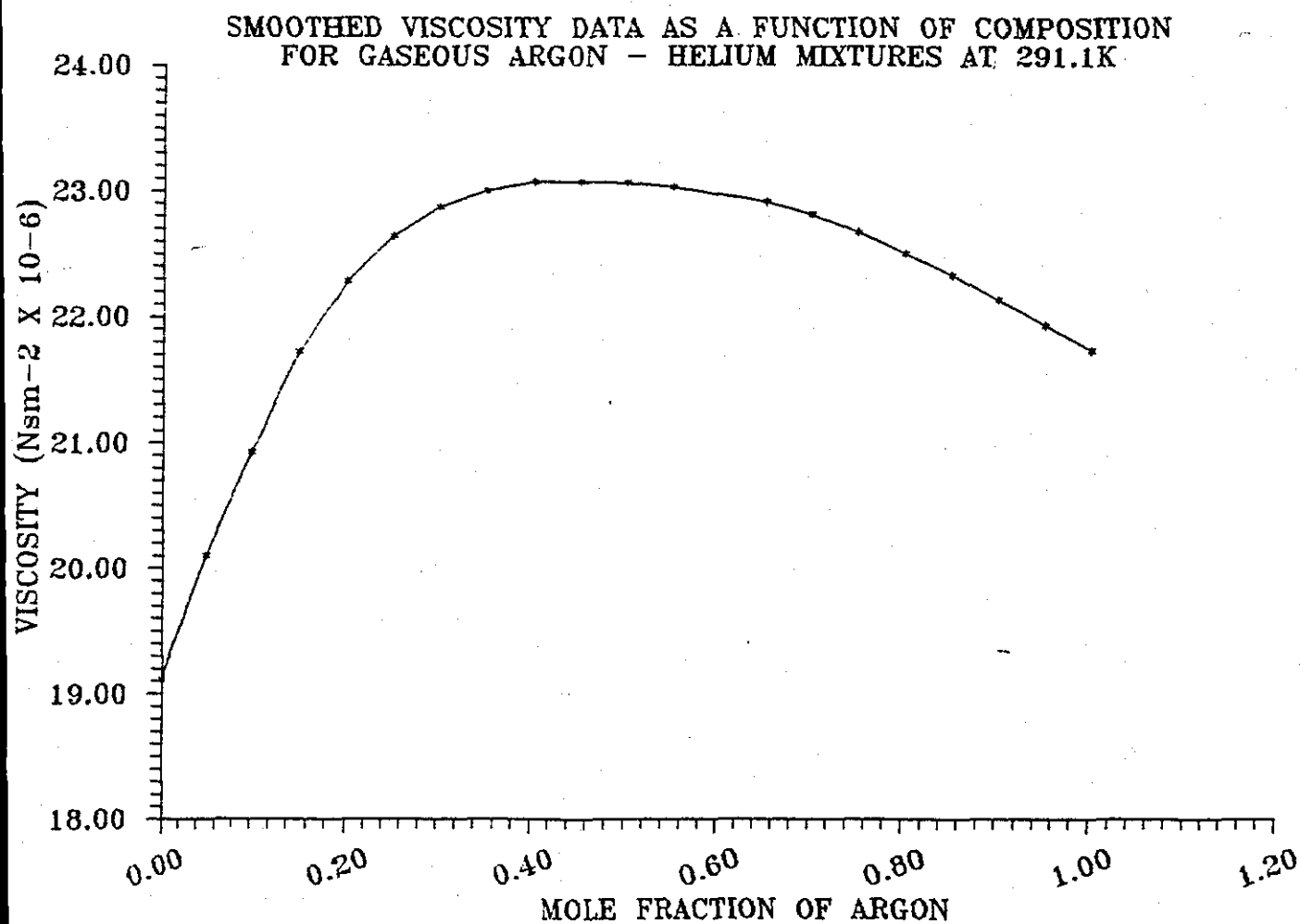


Figure 6.1 Viscosity of Argon/Helium system at 26° C temperature.

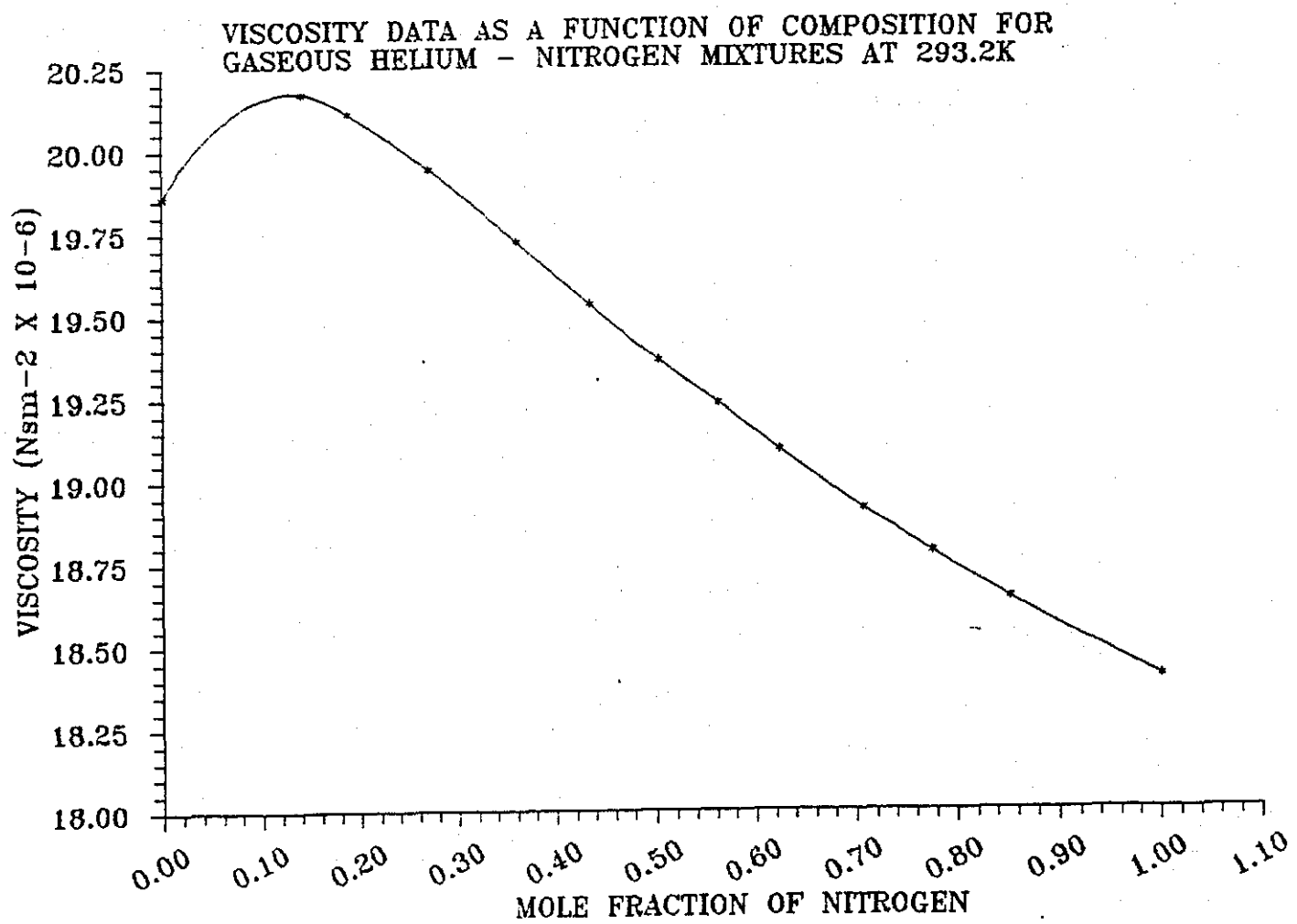


Figure 6.2 Viscosity of nitrogen/helium system at 26° C temperature.

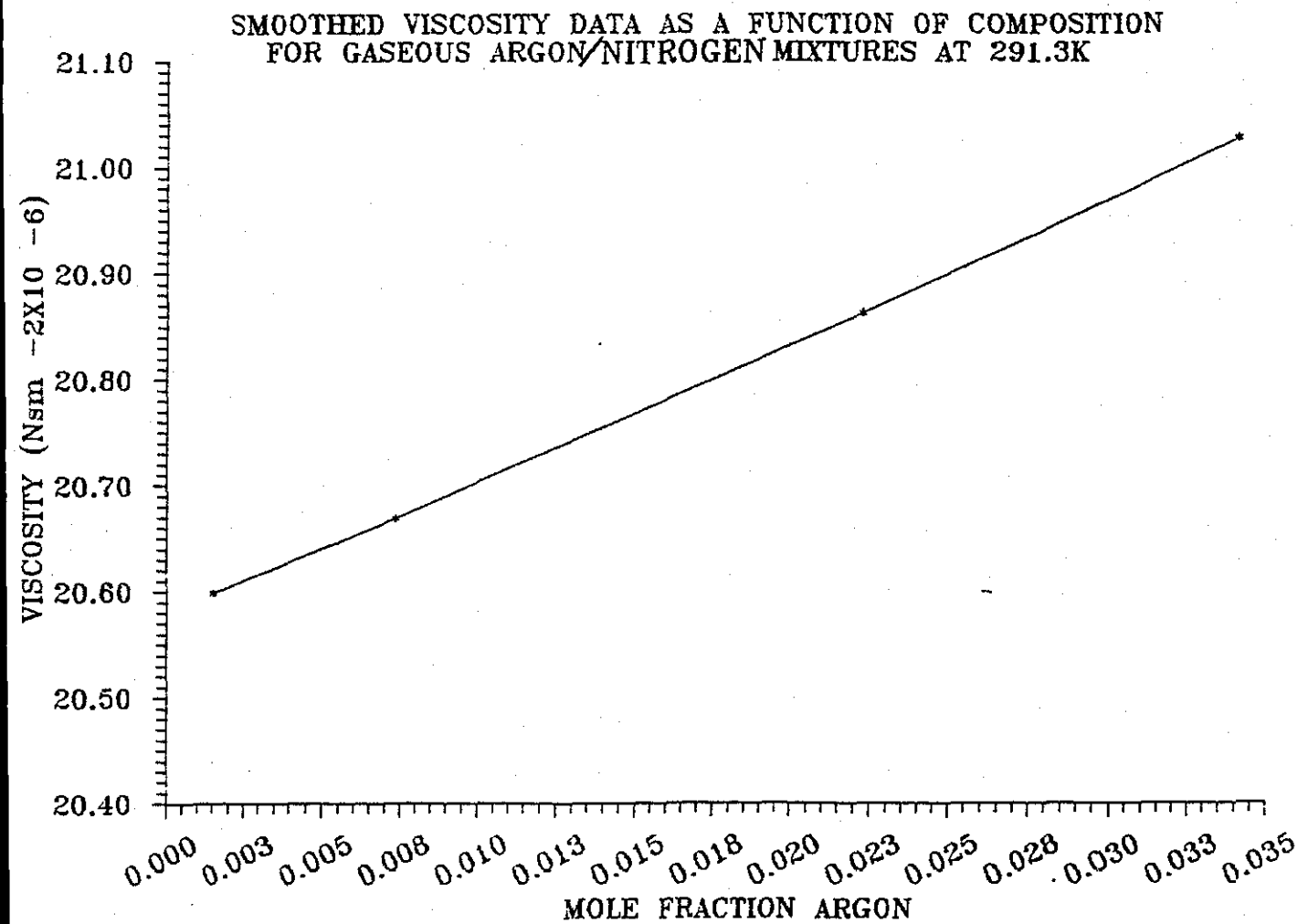


Figure 6.3 Viscosity of argon/nitrogen system at 26° C temperature

## 6.6 The Effect of Pressure On Viscosity Of Pure Gases

The viscosity of gases is a strong function of temperature, but usually a weak function on pressure. The viscosity is a strong function of temperature only in certain regions.

Usually, the effect of pressure on viscosity is not significant at reduced temperatures or low pressures. Figure 6.4 shows the viscosities of Ar, N<sub>2</sub>, and He gases at low pressure and low temperature (room temperature of 20 - 25 °C).

## 6.7 The Effect Of Pressure Gradient

In a packed chromatographic column, the flows in the narrow interstices between the particles of packing, offer resistance to flow, and thus, because of the finite viscosity of the gas, there is a pressure gradient along the length of the column. Such a pressure would have no effect on retention volumes and retention times were it not for the fact that the gas is compressible. The higher pressure at the inlet means that the density of the gas is greater at the inlet than at the outlet. Since the number of molecules per second flowing past a given point at the inlet must be the same as that flowing past a point at the outlet, it follows that the volume flowrate is greater at the outlet than the inlet. Thus, a velocity gradient is an inevitable consequence of the pressure gradient in the column. These two gradients have several consequences in gas chromatography, one of which is to affect the measurement of retention volumes. Normally, the flowrate is measured at the column outlet, so that the measured flowrate is greater than the flowrate at any other point in the column.

Figure 6.5 shows the variation of flow rate observed at the column outlet during chromatography as reported by Conder [30]. A measured retention volume obtained by multiplying the observed retention time by the outlet flowrate is greater than the true retention volume.

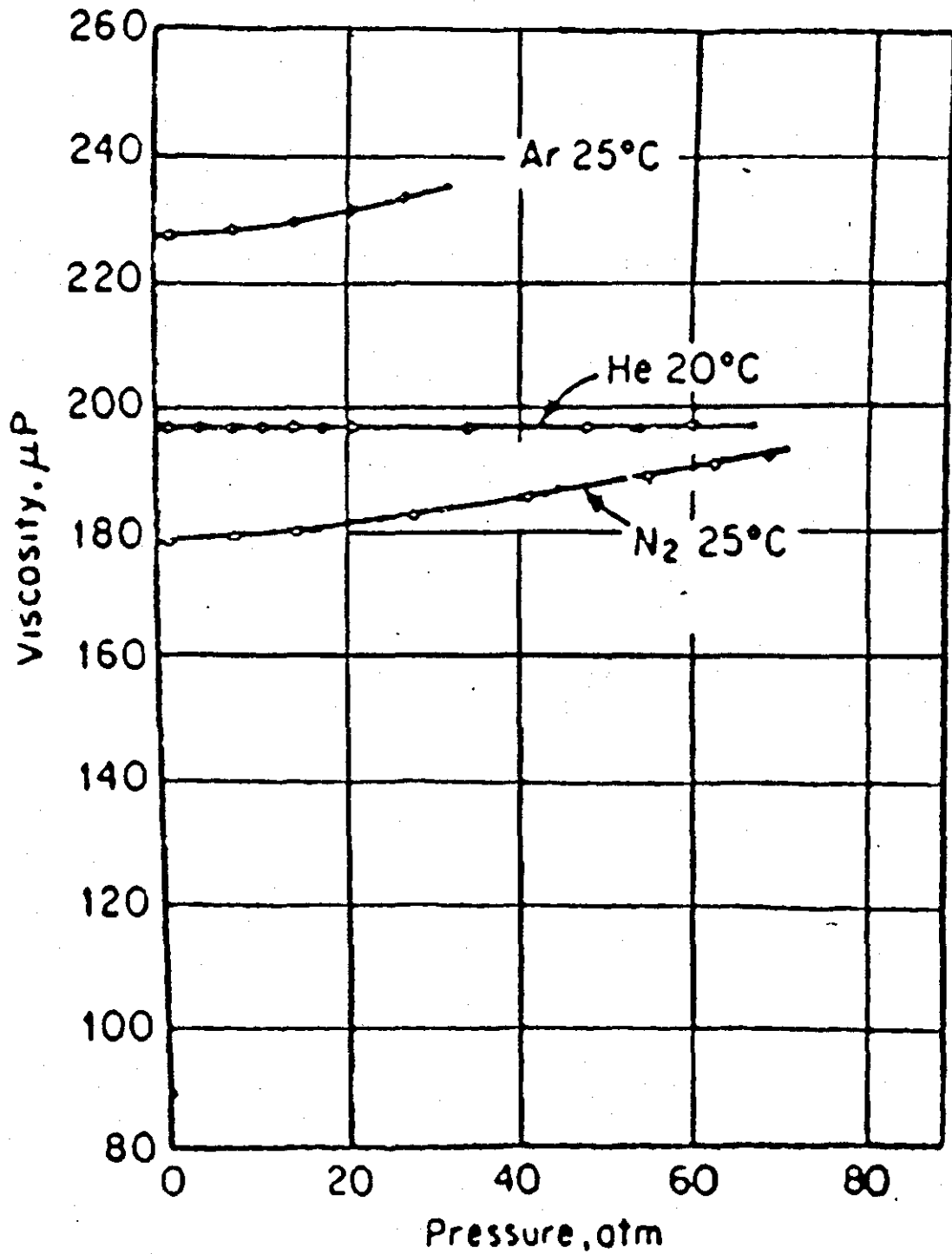


Figure 6.4 show a viscosity of pure gases of argon/helium/ and nitrogen

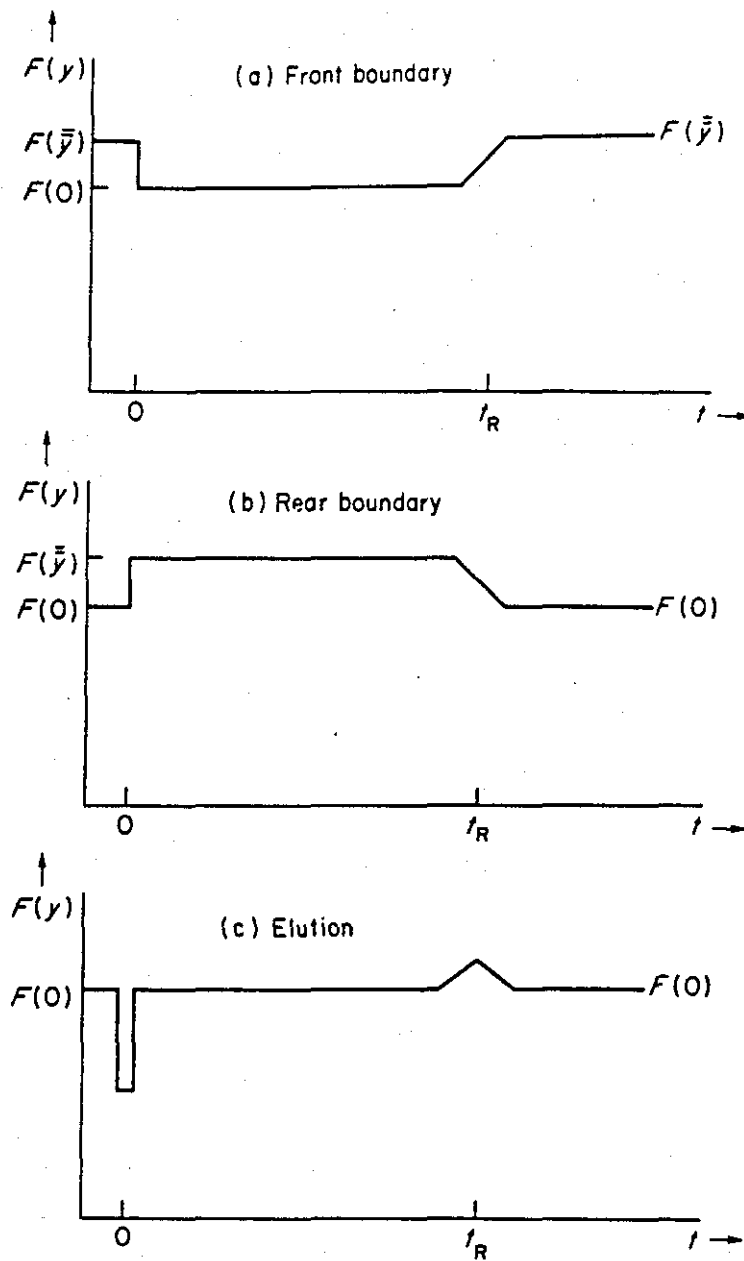


Figure 6.5 show the variation of flow rate  $F$  observed at the outlet column. The boundary is added at time  $t = 0$  and break through at  $t = t_R$ .

## 6.8 Summary Conclusion

The implementation of down stream delay lines enables the effect of viscosity to be separated from the effect of flow rate. Furthermore they enable a viscosity factor to be directly measured from the flowrate chromatogram by simply dividing the offset produced by viscosity by the offset produced by flow rate. These measured viscosity factors can be used to try and predict the entire mixture viscosity curve using only the two measured viscosity factors..

The two end points area (see Chapter 7.3) are a convenient choice because they can be produced easily. Alternatively, one or more mixture points can be employed because this may provide better prediction.

Using the flow rate measurements at the end points, the isotherm gradients can be calculated and can be used to investigate the system. The argon, helium systems, show that the isotherms are nearly straight lines because the gradients are the same at each end.



## CHAPTER SEVEN

BINARY ADSORPTION ISOTHERM GRADIENTS

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## 7.1 Introduction

This chapter describes the procedure used for the measurement of binary mixture isotherms for the argon-nitrogen and helium-nitrogen system. Only adsorption of a single component with a small quantity of another was measured. The adsorbent was Linde 5A molecular sieve pellets at a gas pressure of 1.25 bar and temperature of 60°. The calculation procedures to obtain the binary mixture isotherms data are described and the basic experimental results are presented.

For single-component systems the chromatographic method is simple and straight-forward. The measurement of binary isotherms by equilibrium methods is complex, since simultaneous measurements of both gas-phase and adsorbed-phase composition are required. Therefore for binary systems the chromatographic method can be simpler and quicker, making it ideal for preliminary adsorbent screening.

The earliest work on adsorption isotherms was carried out in 1947 when Gluekauf [31] pointed out that adsorption isotherms could be determined from the break-through curves of gas-solid chromatography. The method has been improved by Conder and Purnell in 1967. Further work has been carried out by Ruthven and Kumar [32] who studied the adsorption of pure and binary gas systems using a concentration step technique. They analysed their results by the procedure suggested by Van de Ulist and Van der Meijden [33]. Another method by Mason Buffham, and Yadav [34] measured the flowrate transient and the composition transient. Later, Mason and Buffham [35] modified the method to make it easier to use than the previous one.

In this research the apparatus configuration was virtually the same as in previous experimental designs

[36]. The column was now fully packed with molecular sieves and had no voids. The Katharometer was a standard Pye semi-diffusion type and was fitted outside the Pye Oven to prevent noise and vibration caused by the fan circulation. Only single components were used as carrier gas and not mixtures across the entire composition range, and so it was only possible to obtain the isotherm gradients at the two end points. The results have been presented graphically in Figure 7.3. Measurements were taken from the flowrate record using a planimeter.

## 7.2 The Theoretical Expressions of Isotherm

In the past a wide variety of theoretical methods and apparatus have been used to determine a desorption isotherm equilibria of pure gases or gas mixtures. The mathematical theory reported here was specially developed by Mason and Buffham as a procedure to calculate the isotherm gradients.

In the initial state of the chromatographic column shown in Figure 7.1, let species  $i$  enters the column at a time,  $t < 0$  and a molar flowrate of  $M_i$ .

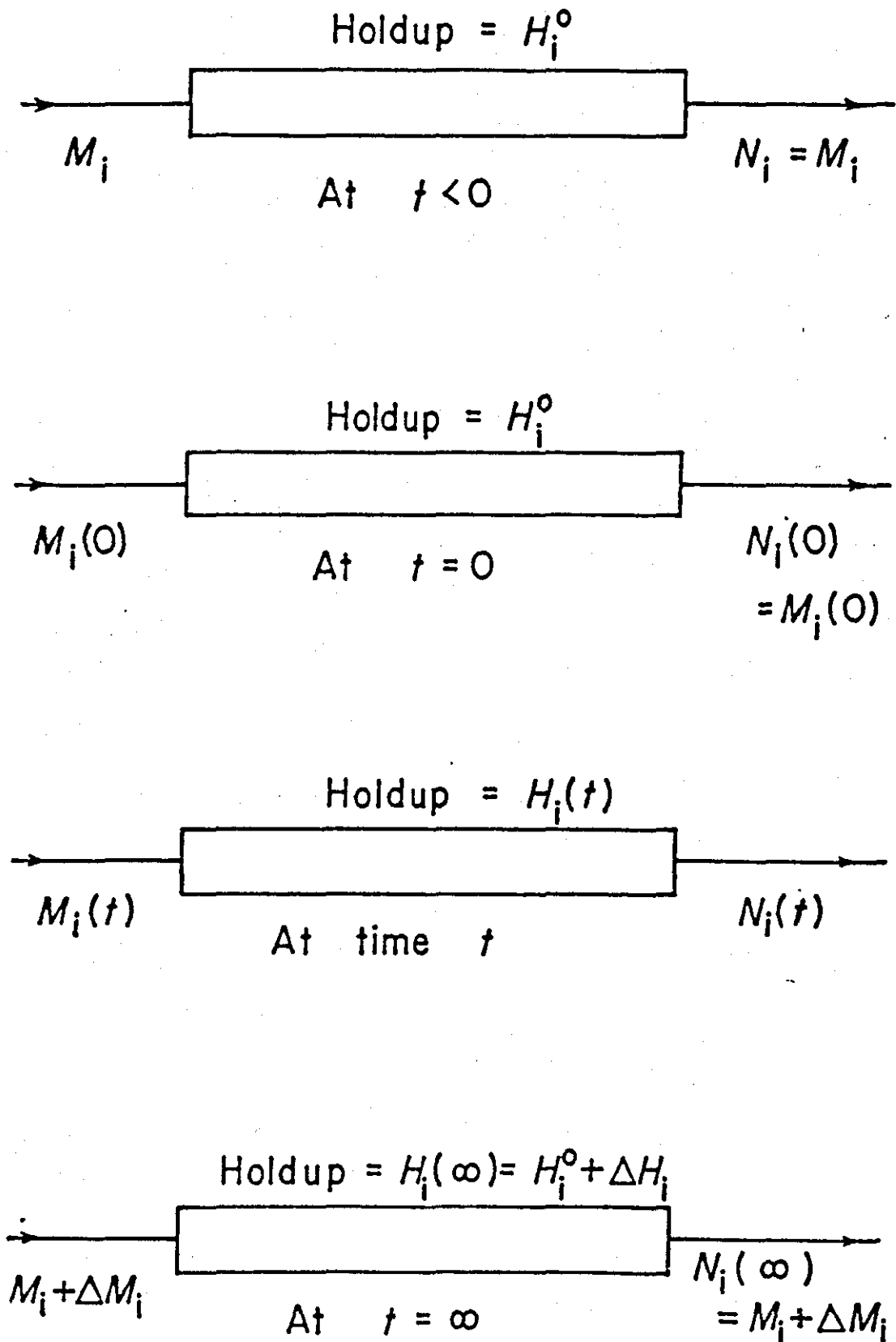


Figure 7.1 The initial state of the column

Let also the flowrate of species  $i$  leaving the column be  $N_i$ . The total molar flowrate of material leaving the column is  $N^0$ . This has a mole fraction  $X_i^0$  of the  $i$ th species.

As the initial state of the column is an equilibrium state, the total amount of material entering the column  $M_i$  equals the total amount leaving the column  $N^0 X_i$ . The molar amount of adsorbent species  $i$  in the column is the hold-up  $H_i^0$ . Now at  $t = 0$  the flowrate of species  $i$  entering the column is changed so that at time  $t$  the molar flowrate of species  $i$  entering becomes  $M_i(t)$ . Let the flow of species  $i$  leaving at time  $t$  be  $N_i(t)$  and the hold-up of species  $i$  at  $t$  be  $H_i(t)$ . The material balance along the column gives:

$$H_i(t) - H_i^0 = \int_0^t M_i(t) dt - \int_0^t N_i(t) dt = \int_0^t [M_i(t) - N_i(t)] dt \quad (7.1)$$

When  $t = \infty$  the column will reach a new equilibrium state. Then the molar flowrate species  $i$  into the column at  $t = \infty$  will be  $M_i + \Delta M_i$ . Let the change in total hold-up of species  $i$  in the column between the initial and final equilibrium states be  $\Delta H_i$ , so that:

$$H_i(\infty) - H_i^0 = \Delta H_i = \int_0^{\infty} [M_i(t) - N_i(t)] dt \quad (7.2)$$

However, when small changes between two equilibrium states are made and  $\Delta M_i$  is small it is convenient to work in terms of perturbation variables. These variables are measured from the initial state at  $t < 0$ . These perturbation variables are  $n(t)$  for the flowrate and  $x_i(t)$  for the mole fraction of species  $i$ . Both of these variables can be experimentally determined. The flow of species  $i$  leaving the column is given by:

$$N_i(t) = (N^0 + n(t))(X_i^0 + x_i(t)) \quad (7.3)$$

Multiplying out and ignoring the cross term  $n(t)x_i(t)$  as being second order and negligible for small perturbations:

$$N_i(t) = N^0 X_i^0 + X_i^0 n(t) + N^0 x_i(t) \quad (7.4)$$

From equation 7.2 consider a step change of  $\Delta M_i$  at  $t = 0$ , so therefore  $M_i(t)$  becomes  $M_i + \Delta M_i$ . Substituting in 7.4 equation for  $M_i(t)$  and  $N_i(t)$  gives:

$$\Delta H_i = \int_0^{\infty} [M_i + \Delta M_i - N^0 X_i - X_i^0 n(t) - N^0 x_i(t)] dt \quad (7.5)$$

$M_i$  equals  $N^0 X_i$  and  $\Delta M_i$  is given by

$$\Delta M_i = N^0 x_i(\infty) + X_i^0 n(\infty) \quad (7.6)$$

gives

$$\Delta H_i = N^0 \int_0^{\infty} [x_i(\infty) - x_i(t)] dt + X_i^0 \int_0^{\infty} [n(\infty) - n(t)] dt \quad (7.7)$$

If we sum  $\Delta H_i$  over all species we obtain

$$\sum_i \Delta H_i = N^0 \int_0^{\infty} 0 dt + \int_0^{\infty} [n(\infty) - n(t)] dt = \int_0^{\infty} [n(\infty) - n(t)] dt \quad (7.8)$$

The change in the total hold-up in the column is the integral of difference between  $(n(\infty))$  and  $(n(t))$ . If the chromatographic column has volume  $V$  and is packed with an adsorbent to a porosity  $\varepsilon$  the volume of packing will be  $V\varepsilon$  and the void volume  $V(1-\varepsilon)$ . In the transition between initial and final equilibrium states the change in concentration of species  $i$  is  $\delta c_i$  in the void space and in  $\delta q_i$  the adsorbent so that a material balance gives the change in hold-up in the column :

$$\Delta H_i = \varepsilon V \delta c_i + (1 - \varepsilon) V \delta q_i \quad (7.9)$$

By rearranging the above equation the isotherm gradient is:

$$\frac{\delta q_i}{\delta c_i} = \frac{\frac{\Delta H_i}{\delta c_i} - \varepsilon V}{(1 - \varepsilon) V} \quad (7.10)$$

The volume of packed bed and  $\epsilon$  is given by:-

$$\begin{aligned}\text{volume of packed bed } V &= \pi/4 d_i^2 L \\ &= \pi/4 \times (0.4)^2 \times 150 \\ &= 18.85 \text{ cm}^2\end{aligned}$$

$\epsilon$  porosity

$$\begin{aligned}&= \frac{\text{volume of space}}{\text{total volume}} \\ &= 0.376\end{aligned}$$

The change in the concentration of species  $i$  in the adsorbent is caused by the change of concentration gas in the void space  $\delta q_i / \delta c_i$ . This represents a gradient of the equilibrium surface given by  $q_i$  against  $c_i$ .  $\delta q_i / \delta c_i$  was determined from the change in hold-up of  $i$  with concentration,  $\delta H_i / \delta c_i$ . The step change in  $M_i$  is made at  $t = 0$ . In an experiment the trace flow used to perturb the concentration is usually a pure stream of one of the components. But here we consider the general stream of composition  $X_i^T$  of species  $i$ . The flowrate is  $n(\infty) - n(0)$  which is term  $n$ . The flowrate of added species  $i$ ,  $\Delta M_i$  is:

$$\Delta M_i = n X_i^T \quad (7.11)$$

If the density of the gas in the column ( $\rho$ ) is unchanged by addition of stream  $n$  then the change in concentration of species  $i$  in the void space  $\delta c_i$  is given by:

$$\delta c_i = \rho [x_i(\infty) - x_i(0)] \quad (7.12)$$

Re-arranging (7.12)

$$\frac{\delta c_i}{\rho} = x_i(\infty) - x_i(0) \quad (7.13)$$

From equation 12, the r.h.s is the difference in mole fraction of species  $i$  between time zero and time when equilibrium is reestablished in the new position (steady

state). Using the main flowrates this change can be expressed:

$$x_i(\infty) - x_i(0) = \frac{MX_i^0 + nX_i^T}{M + n} - X_i^0 \quad (7.14)$$

When  $M$  is the total molar flowrate entering the column at  $t = 0$ , gives:

$$x_i(\infty) - x_i(0) = \frac{n[X_i^T - X_i^0]}{M + n} \quad (7.15)$$

But  $n$  equal  $n(\infty) - n(0)$ , so therefore:

$$\delta c_i = \frac{Q(n(\infty) - n(0))(X_i^T - X_i^0)}{M + n} \quad (7.16)$$

Now dividing equation 7.7 by  $\delta c_i$  and using 7.12 and 7.16 to express  $\delta c_i$  in the first and second terms on the r.h.s of equation gives:

$$\frac{\Delta H_i}{\delta c_i} = \frac{N_0}{Q} \int_0^\infty \frac{x_i(\infty) - x_i(t)}{x_i(\infty) - x_i(0)} dt + \frac{X_i^0(M + n)}{Q(X_i^T - X_i^0)} \int_0^\infty \frac{n(\infty) - n(t)}{n(\infty) - n(0)} dt \quad (7.17)$$

Let the gas leaving the chromatographic column at atmospheric pressure  $P_{at}$  and the pressure drop down the column be  $\Delta P_0$ . If  $\Delta P_0$  is small relative to  $P_{at}$  then the mean pressure in the column is  $(P_{at} + 1/2\Delta P_0)$ . If the gas flowrate leaving the column is measured as  $Q_{at}$  at pressure  $P_{at}$ , and temperature  $T_{at}$ , with the column temperature  $T$  then the ideal gas equation gives:

$$\frac{N_0}{Q} = Q_{at} \frac{P_{at}}{(P_{at} + 1/2\Delta P_0)} \frac{T}{T_{at}} \quad (7.18)$$

$$N_0 = (M+n) \quad (7.19)$$

Substituting 7.18 into 7.17 gives

$$\frac{\Delta H_i}{\delta c_i} = \frac{Q_{at} P_{at}}{(P_{at} + 1/2 \Delta P_o) T_{at}} \left[ \int_0^\infty \frac{x_i(\infty) - x_i(t)}{x_i(\infty) - x_i(0)} dt + \frac{X_i^0}{(X_i^T - X_i^0)} \int_0^\infty \frac{n(\infty) - n(t)}{n(\infty) - n(0)} dt \right] \quad (7.20)$$

It is therefore possible to obtain the gradient of the adsorption isotherms by finding the value of  $\Delta H_i / \delta c_i$  and then substituting this in to equation 7.10. At low concentrations the gradient of the isotherm will be the Henry's law constant.

In order to evaluate  $(\Delta H_i / \delta c_i)$ , the values of  $T_n$  and  $T_x$  are required. These can be found from the chart recorder traces, and involve dividing particular particular areas by particular offsets. Hence, equation 7.21 below is obtained by substituting  $\tau_n$  and  $\tau_x$  in equation 7.20.

$$\frac{\Delta H_i}{\delta c_i} = \frac{Q_{at} P_{at}}{(P_{at} + 1/2 \Delta P_o) T_{at}} \left[ \tau_x + \frac{X_i^0}{X_i^T - X_i^0} \tau_n \right] \quad (7.21)$$

### 7.3 Calculation Procedure

Usually, the isotherm gradient would be measured at different compositions spanning the whole range. These gradients could then be integrated to give the actual binary isotherms. However, with the present experimental set up, no mixtures were used as bulk flows, only the pure components. Therefore, it was only possible to obtain the isotherms gradients at the two end points. Because there are two binaries, a total of four gradients as shown below in Figure 7.2 can be obtained. For a binary mixture, the following mole fraction rules are employed:

$$X_{AR}^o + X_{He}^o = 1$$

$$X_{AR}^T + X_{He}^T = 1$$



where 0 refers to the bulk composition and T refers to the trace flow composition.

Trace A to Pure B  
will give end point  
gradients here.

Trace B to pure A  
will give end point  
gradients here.

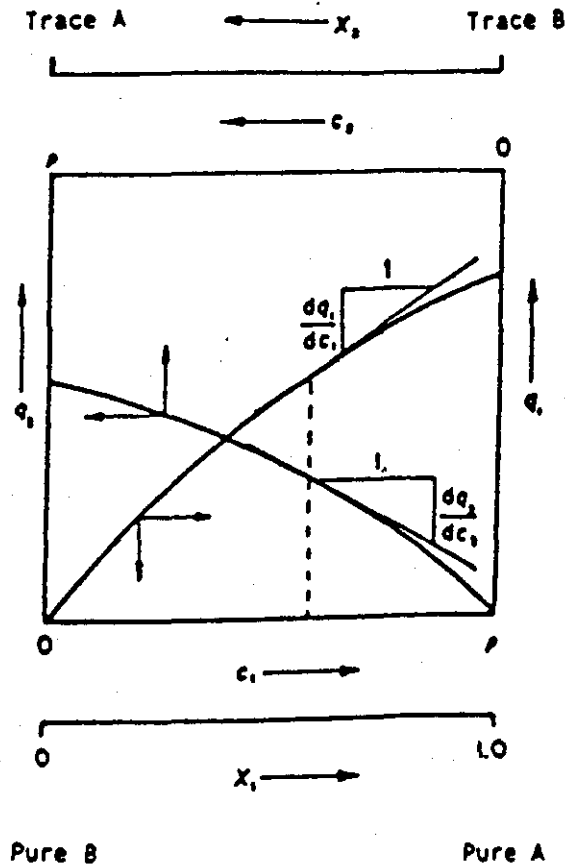


Figure 7.2 Shows the equilibrium curves slopes at infinite dilution.

In previous diagram the end points were shown. Now here is the procedure of calculating these points. For the system with a helium bulk flow and an Argon trace the apparatus set up gives:

$$\text{the flow for both columns} = 32 \text{ ml/min}$$

$$\text{therefore, for one column ml/min to ml/sec.}$$

$$= 32 \times 1/2 \times 1/60 = 0.266 \text{ ml/sec.}$$

$$\text{pressure } P_{at} = 1.0 \text{ bar}$$

$$\begin{aligned} \text{column mid point pressure} &= P_{at} + 1/2 \Delta P_o \\ &= 1.05 \end{aligned}$$

$$\text{Temperature} = 60 + 273 = 333$$

$$T_{at} = 20 + 273 = 293$$

$$\text{column mid point flowrate} = P_{at} + (R_1 + R_2) / 2$$

$$\begin{aligned} Q_c &= 0.266 \times 1/1.05 \times 333/293 \\ &= 0.287 \text{ ml/sec.} \end{aligned}$$

$$\text{volume of the column} = 18.85 \text{ cm}^3$$

$$\text{Voidage} = 0.376$$

$$\tau_x = 19 \text{ min} = 19/30 \times 60 \text{ sec} = 38 \text{ sec}$$

$$\text{offset} = 3.73 \text{ cm}$$

$$\text{area} = 1/2 \times 6 \text{ cm}$$

$$\begin{aligned} \tau_n &= \text{area}/\text{offset} \\ &= 3.73 \times 3 \\ &= 11.19 \text{ cm} \end{aligned}$$

Using equation 7.21 for the helium gradient;  $i = \text{He}$ ,  $X_i^o = 1$  and  $X_i^T = 0$ .

$$\begin{aligned} \Delta H_i / \delta C_i &= 0.287 [ 38 + (11.19) (-1) ] \\ &= 7.7 \text{ ml/sec.} \end{aligned}$$

Using equation 7.21 for the argon gradient;  $i = \text{Ar}$ ,  
 $X_i^0 = 0$  and  $X_i^T = 1$ .

$$= 0.287 [ (38) + (11.19) (0) ]$$

$$= 14.1 \text{ ml/sec.}$$

sub. in equation 7.10 where column volume =  $18.85 \text{ cm}^3$  and  $\epsilon$   
 $= 0.376$  for helium and argon. This gives for pure helium:-

(1)

$$\frac{\delta q_i}{\delta c_i} = \frac{7.7 - (0.376 \times 18.85)}{(1 - 0.376) \times 18.85}$$

The isotherm gradient for helium = 0.6

(2)

$$\frac{\delta q_i}{\delta c_i} = \frac{10.9 - 7.0876}{11.7624}$$

The isotherm gradient for argon = 0.32

Using equation 7.21 for the system with argon bulk-flow and helium perturbation flow substitute for argon first and helium second. As the temperature and pressure of the system is constant. Therefore, the pressure at column mid point is the same as in previous system.

The total columns flow = 36 ml/min.

therefore, for one column =  $36 \times 1/2 \times 1/60$

= 0.3 ml/sec.

column mid point flowrate

$$= P_{at} + \frac{(R1 + R2)}{2}$$

$$Q_c = 0.3 \times 1/1.05 \times 333/29$$

$$= 0.325 \text{ ml/sec}$$

Using equation 7.21 for the argon gradient;  $i=\text{Ar}$ ,  $X_i^0 = 1$  and  $X_i^T = 0$  to find end point 1.

$$(1) \quad \text{Ar} = 0.325 \text{ ml/sec}$$

$$\Delta H_i / \delta C_i = 29.25 \text{ ml}$$

Using equation 7.21 for the helium gradient;  $i=\text{He}$ ,  $X_i^0=0$  and  $X_i=1$  to find end point 2.

$$(2) \quad \text{He} = 0.325 \text{ ml/sec}$$

$$\Delta H_i / \delta C_i = 13.13 \text{ ml}$$

substitute in equation 7.10 for argon and helium trace to find end point 3 and 4.

$$\begin{aligned} \frac{\delta q_i}{\delta c_i} &= \frac{29.25 - (0.376 \times 18.85)}{(1 - 0.376) \times 18.85} \\ &= 1.884 \end{aligned}$$

$$\begin{aligned} \frac{\delta q_i}{\delta c_i} &= \frac{13.13 - 7.0876}{29.25} \\ &= 0.513 \end{aligned}$$

Comparing each pair of gradients (1.2 and 1.88, and 0.32 and 0.51) it can be seen that the gradients at each end of the composition range are approximately the same and so the two isotherms are nearly straight lines. Figure 7.3 shows both isotherm gradients plotted against composition.

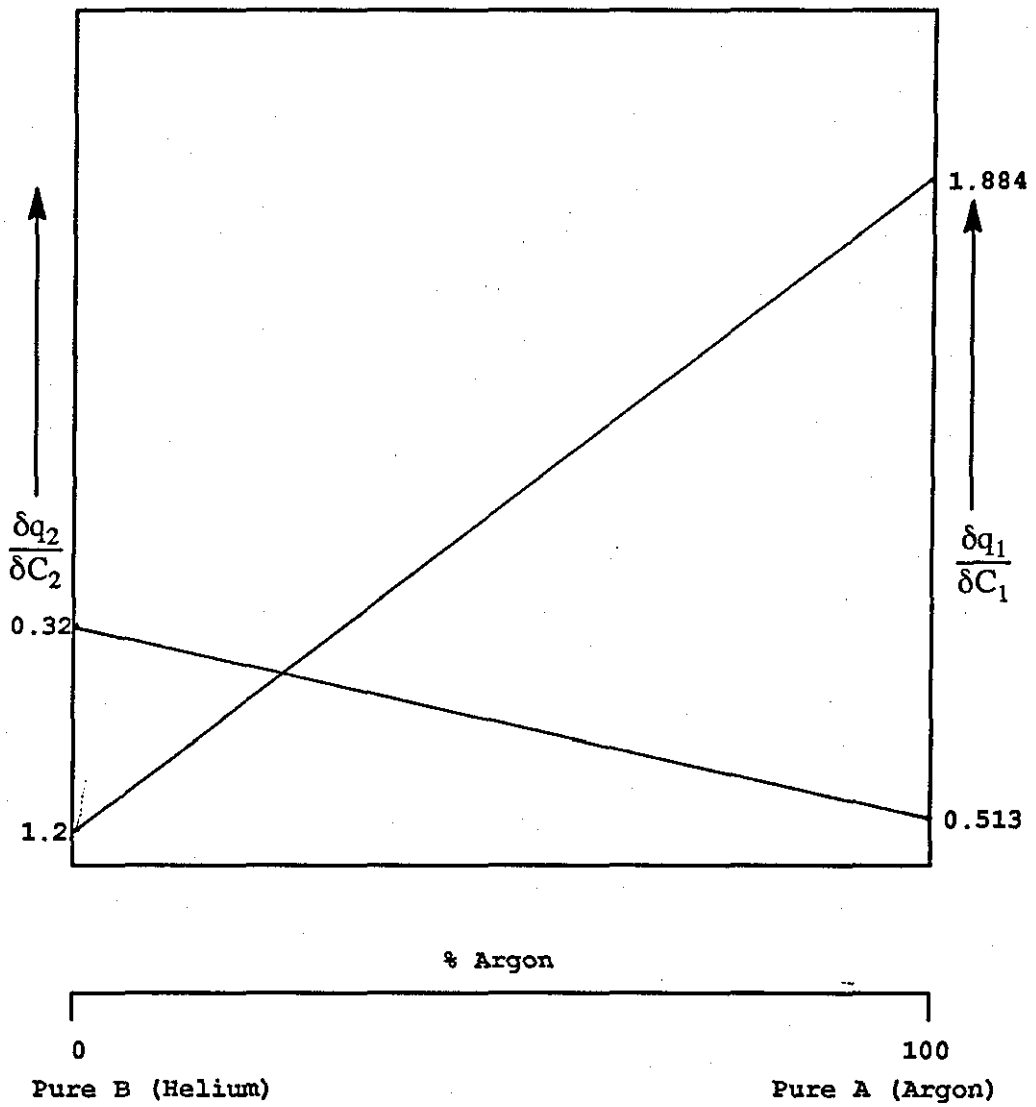


Figure 7.3 The isotherm gradients diagram.

## CHAPTER EIGHT

## RESULTS

The results obtained in this chapter are based on operating the system with and without downstream delay lines. Also reported here is the detection of a void in packed chromatographic columns using carrier and small trace flows of a tracer gas.

### 8.1 Experiment Configuration One

For the experiments reported here the chromatograph was fitted with two glass columns each of 4 mm internal diameter and 1.5 m long. One column had a 60 mm glass-wool void (located 72 cm from either end) within the 500-710  $\mu$ m size Linde 5A molecular sieve packing; the other column was uniformly packed and was used as a reference.

Constant flow-rates of the carrier gas to both columns were maintained by means of a single Porter pressure regulator and a pair of thermostatted matched capillary chokes and two trimming needle valves. One three metre length of 3 mm o.d. nylon tubing (delay line) was connected downstream of the perturbation trace mixing device (TMD) and upstream of the inlet of the void column. A further 10 m of delay line (Nylon tubing) with approximately the same internal volume as an empty column was attached between the TCD and the capillary flow restrictors in both side of the system. The outlets of the columns were connected to the Katharometer cell (TCD).

A differential pressure transducer (DPT) (Furness FCO 40  $\pm$  0.5 mm water gauge) was connected upstream of the restrictors in order to detect the difference between the flowrates. The ratio of the pressure drop across the upstream choke, the column and the downstream restrictors was about 120 : 40 : 1 at the experimental flowrate. The outlet of the matched restrictors was connected to the modified back pressure regulator (BPR). The downstream arrangement of a differential transducer and matched restrictors acts as a very sensitive differential flow meter

and is easily able to measure flowrate fluctuations of about one part in ten thousand of the overall flowrate. At the same time, the overall flowrates in the columns are determined mainly by the upstream chokes. The role of the upstream chokes and downstream restrictors are quite distinct: the upstream chokes main purpose is to set the inlet flow, whereas the downstream restrictors senses small changes in the outlet flow.

A two-pen potentiometric recorder (PM 8252) was used to record the differential-flow-meter reading alongside the output from the conventional diffusion-fed katharometer (Pye). The pressure transducer used in the differential flow meter had a sensitivity of 0.5 volt for full-scale deflection.

The katharometer bridge current was set at 10 mA and the signal for the standard katharometer control was fed to the second recorder channel, which was set at 5 mV full scale deflection. The two channels of the recorder were switched to operate in opposite senses to permit the katharometer and flow-detector trace to be compared directly. Typical data obtained from the chart-recorder are shown in the following figures.

Figure 8.1 helium was used as carrier gas at a flowrate of 40 mL/minute at 1.25 bar. A 0.5% nitrogen perturbation flow (0.2 mL/min) was added upstream of the column with the void, and the response of the pressure transducer was monitored on a potentiometric chart recorder at a speed of 30 mm/min.

When the pertubation flow of nitrogen is added, the chart-recorder shows an increase in flow. Because of the upstream delay line, the recorder trace will level out on a plateau because the nitrogen takes time to reach the column. When the nitrogen pertubation flow reaches the column, adsorption occurs and this is seen as a pause in flow. As nitrogen reaches the void zone the peak appears as the net result. However when the nitrogen trace reaches the column exit the flow returns to the steady state value. When the nitrogen reaches the exit capillary tube there is a shift in baseline caused by the change in viscosity.

Figure 8.2 shows what happens when a 0.4% argon perturbation flow is added to helium carrier. The resultant graph shows that argon is less strongly adsorbed than nitrogen and travels through the column more rapidly. However, it can be seen that the perturbation flow when traversing the void has given the same shape when adding the small flow as when it is removed.

Figure 8.3 the graph demonstrate the results obtained when adding a 0.5% helium perturbation flow to argon carrier. It also show that the advancing fronts are consistent with the retreating one and a mirror-image response is achieved.

Figure 8.4 shows a typical response of a 0.7% nitrogen tracer flow added to argon carrier gas. In operation this trace is switched to one side of the system, at time zero.

The column outlet flow rate is reduced when sorbable material such as nitrogen enters the packing and is adsorbed by the stationary phase. The net effect of the void zone is to give a surge in flow and to produce a peak relative to the baseline on a recording of flowrate against time. The flow rate increases when the sorbed material reaches the end of the column. These results show the behavior of the flow inside the chromatographic column. Argon is shown to be less strongly adsorbed than nitrogen and travel through the column more rapidly.



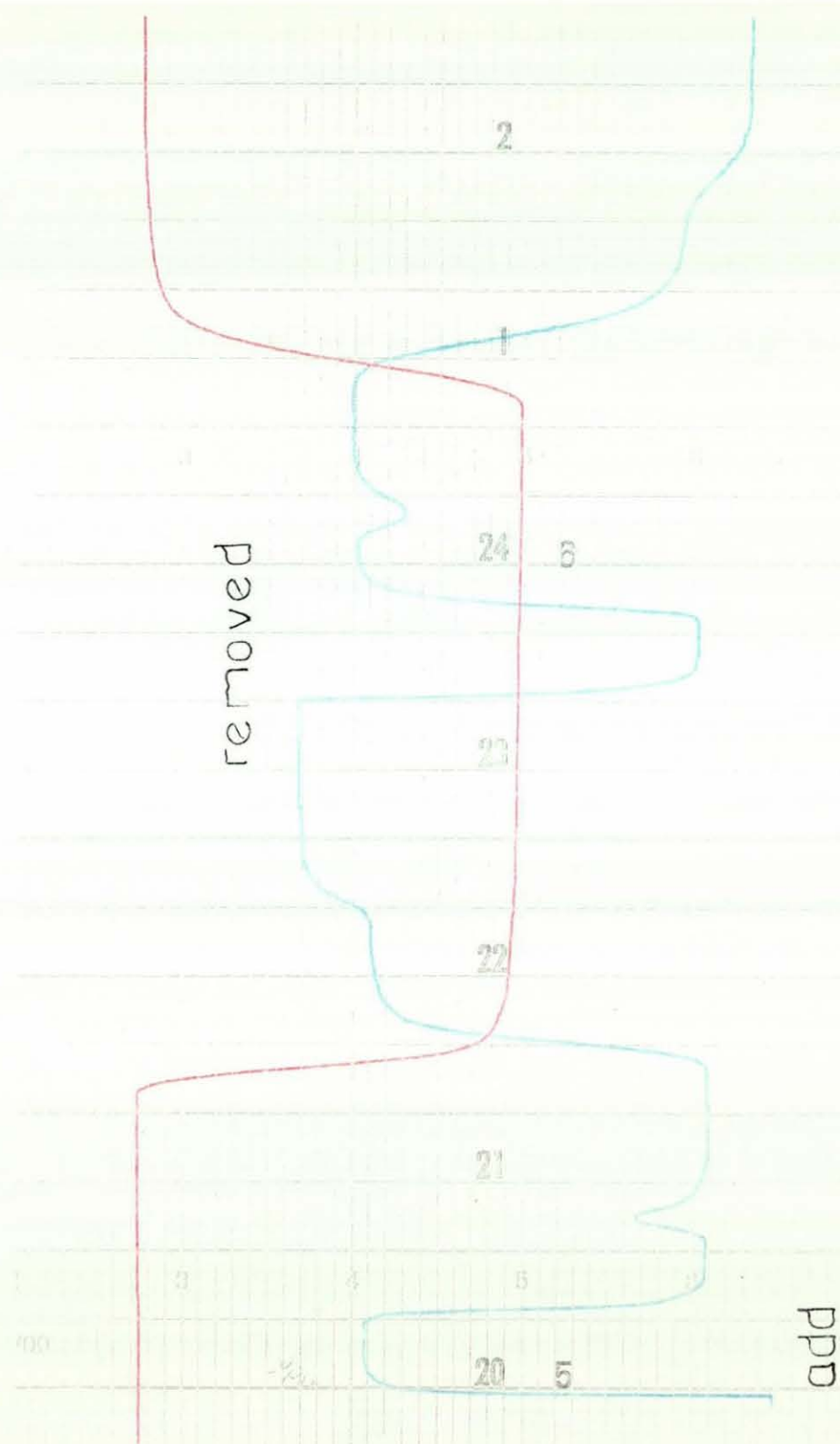


Figure 8.1 show graphically the mirrors image obtained when a 0.5% nitrogen pertubation flow is added to helium bulk flow gas.

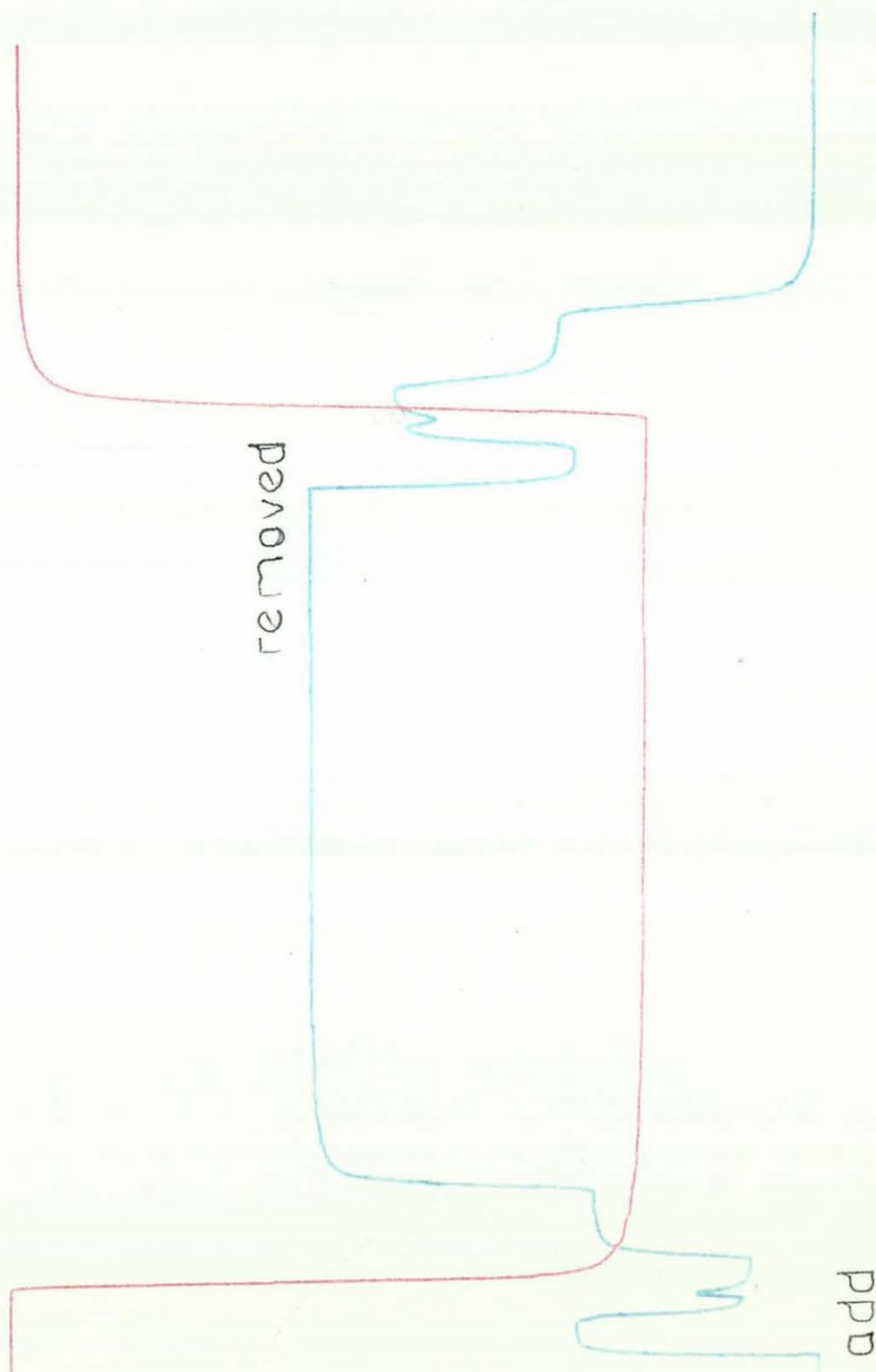


Figure 8.2 ideal chromatograph of void and perturbation flow obtained a 0.4% argon flow added to helium bulk flow gas (carrier).

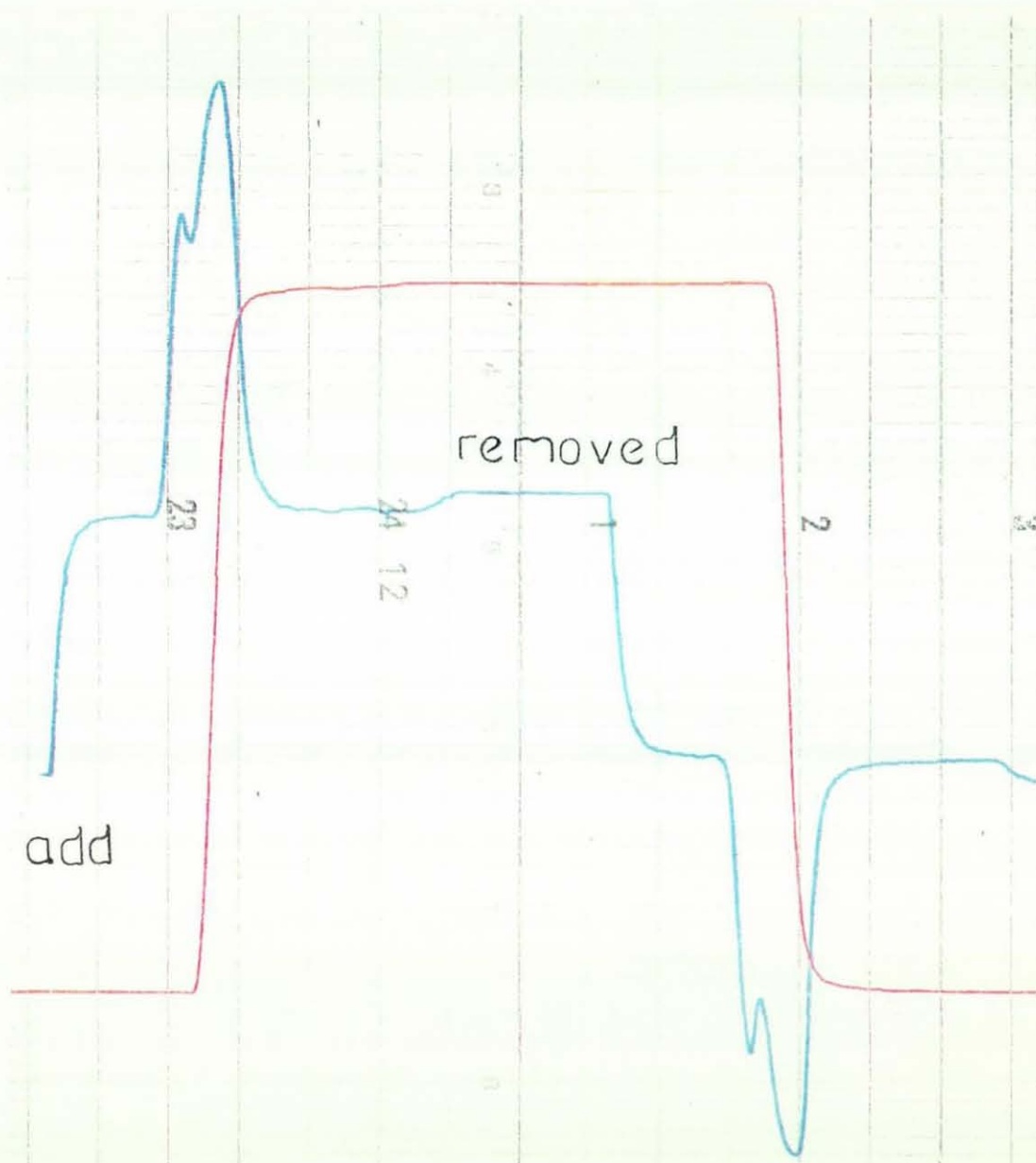


Figure 8.3 Mirror image graph of void and perturbation flow obtained, when a 0.4% helium flow is added to argon carrier gas.

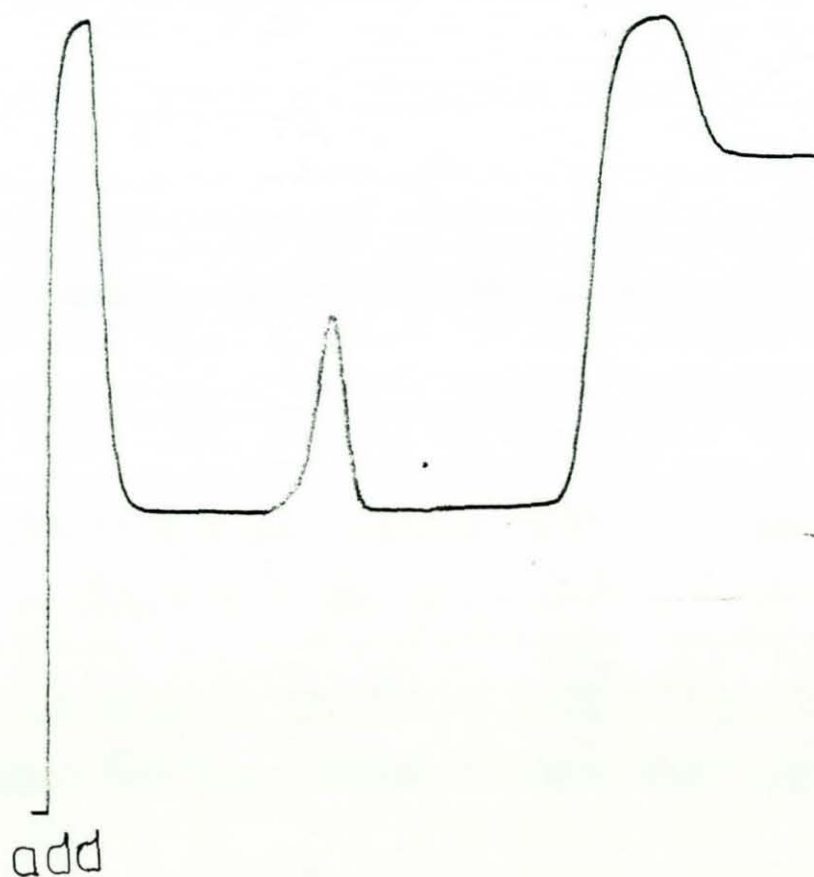


Figure 8.4 show a chromatograph of void and perturbation flow, when a 0.5% nitrogen added to argon carrier gas.



## 8.2 Experiment Configuration Two

The apparatus set-up is similar to that reported in configuration 1, but the columns now contained a short slug of active adsorbent in an inert column rather than an active column with a short slug of non-adsorbent. The column was packed with glass ballotini (beads) 850-1000  $\mu\text{M}$ , and included an active section of length 60 mm, packed with Linde 5A molecular sieve. The chromatograph operated at room temperature of 26°. The results obtained are shown graphically in Figure 8.5 to Figure 8.8. These were: helium - nitrogen, helium - argon, argon - nitrogen, and nitrogen-helium.

Figure 8.5 is an example of the results obtained when a 0.5% nitrogen perturbation flow is added to 36 mL/minutes of helium carrier. The speed of the chart - recorder was set to 30 mm/min and a sensitivity of 0.2 volts. The katharometer sensitivity attenuation was 8 and the chart recorder setting 5mV. These settings were the same throughout the runs in this configurations. Nitrogen is more adsorbed than helium so that when it enters the active section it is adsorbed and when it leaves it is desorbed.

Figure 8.6 The graphic results reported here is the net results of adding 0.4% nitrogen perturbation flow to 36 mL/minutes argon the carrier gas. The chart recorder speed is 30 mm/min, and a sensitivity of 0.2 volts for the flowrate channel. These typical results show a mirror-image response for addition and subtraction of the perturbation trace gas. The response of an active section to the perturbation flow is the inverse of the void overall behaviour. This behaviour is also similar to that in Figure 8.5 except when the front enters the measuring capillaries the effect of viscosity gives a response in the opposite direction.

Figure 8.7 shows the addition and subtraction responses to the perturbation flow when a 1% helium flow is added or removed to 36 ml/minute of nitrogen carrier. The area under the response peak is also the same when the perturbation flow is added to the system and when is removed (switch to vent).

Figure 8.8 shows mirror-image results when a perturbation flow of 0.41% of argon was added to helium carrier gas. The chart-recorder setting speed was 10mm/minutes and a sensitivity of 0.2 Volts for the flow channel.

From the graphs reported in configuration 1 and 2 the apparatus has shown that "mirror-image" responses can be obtained, with clear resolution of sorption/desorption effects. With the modified (BPR) the system is unaffected by noise and pressure fluctuations during the experiments.

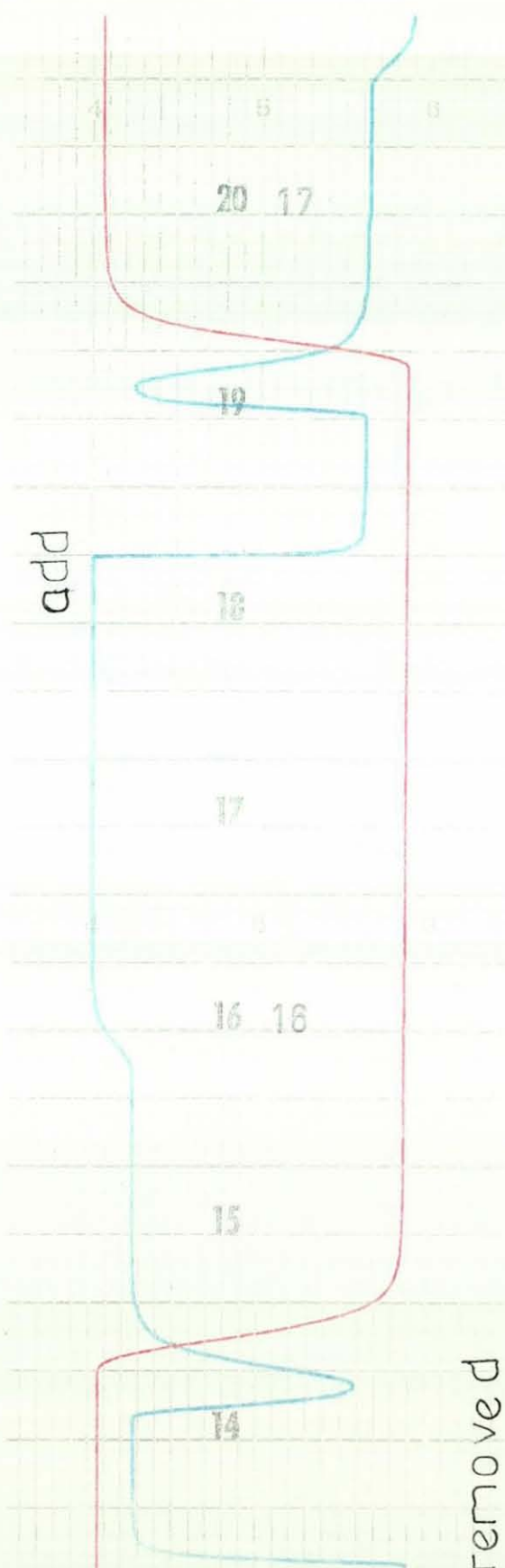


Figure 8.5 shows mirror-image graph of the perturbation flow when a 0.5% nitrogen flow is added to helium carrier.

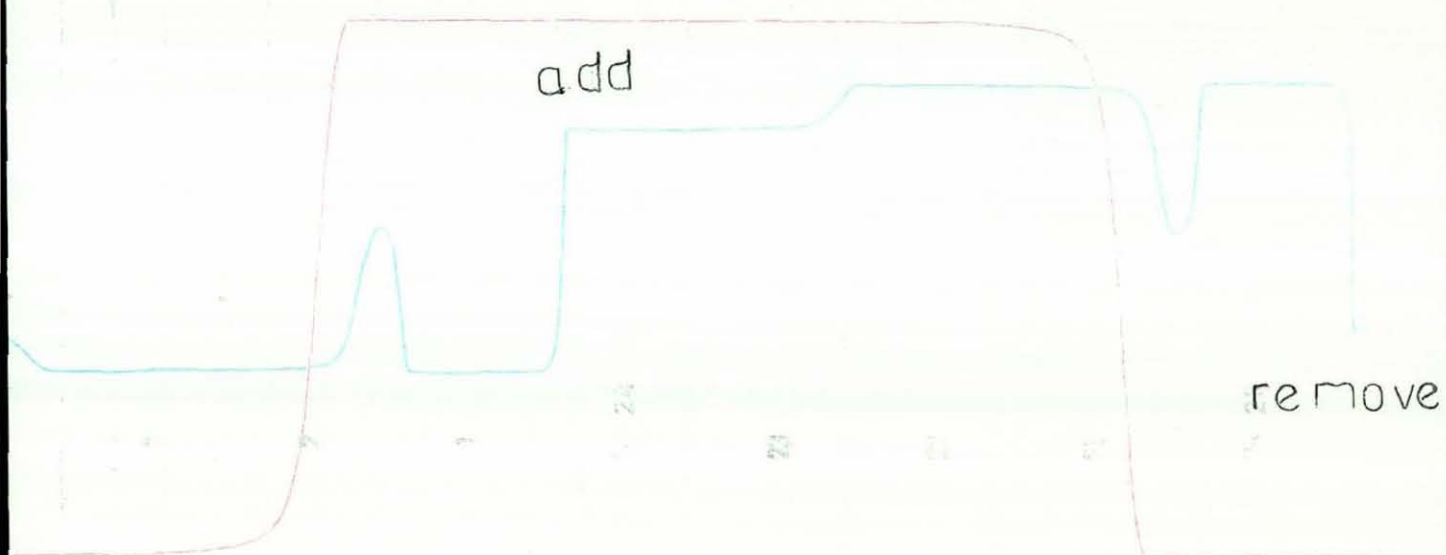


Figure 8.6 shows a mirror-image graph of the perturbation flow when a 0.4% nitrogen flow is added to argon carrier.



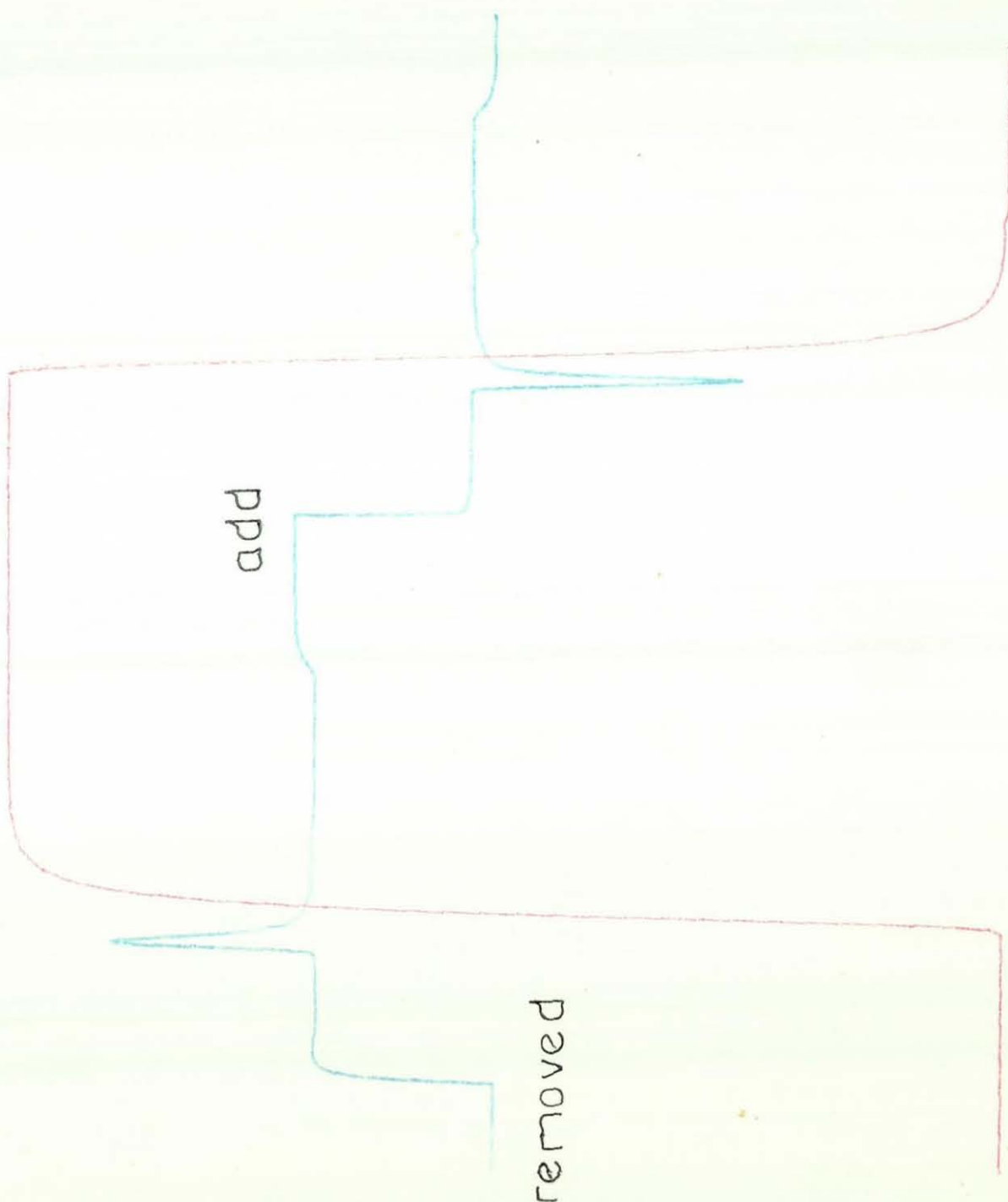


Figure 8.7 shows a mirror image of the resulting graph when a 1% helium perturbation flow is added to nitrogen carrier.

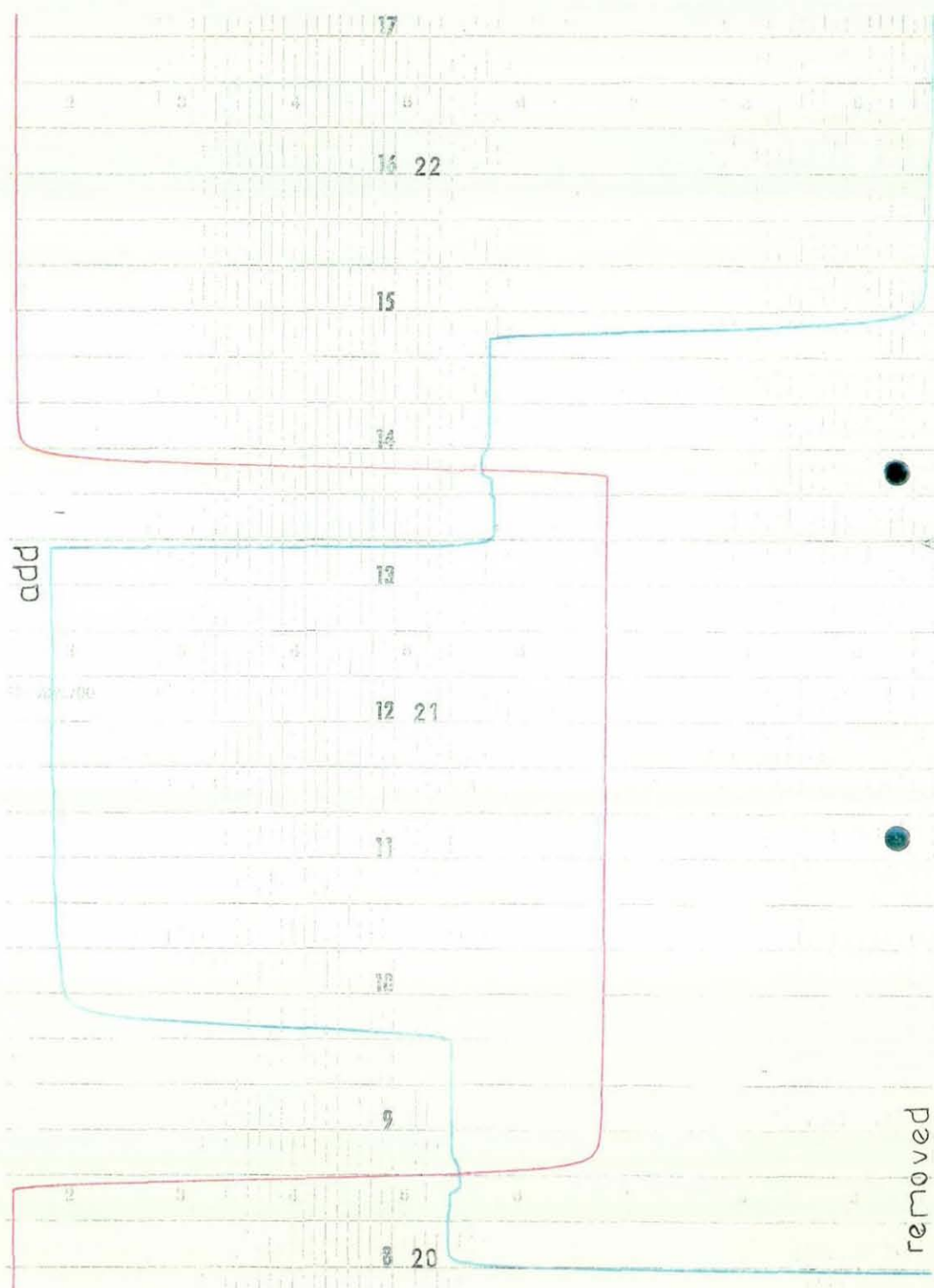


Figure 8.8 shows a mirror image of the resulting graph when a 0.41% argon perturbation flow is added to helium carrier.

### 8.2.1 Calculation Procedure

The theory and the calculation procedure reported here is to demonstrate that the area under the flow rate fluctuation produced by a void in a packed chromatographic column is the same when the perturbation flow is added to the column as when it is removed (switched to vent). The shapes of the flowrate fluctuation may differ (i.e not be mirror-images) but the areas should be the same. Since the perturbation trace flow is effectively cut off completely when is switched to vent, the perturbation flow transient should be a mirror-image an ideal addition transient. As we have seen before, it is difficult to make the addition and subtraction perturbations identical. From the chart-recorder response of the deviation of the flowrate caused by the desorption and resorption at the void the area under the peak to be calculated and the volume of the void determined.

Theory suggests that area 1 under peak 1 and area 2 under peak 2 should be the same (see Figure 8.1). This is confirmed by experiment. The void volume was estimated to be within 20% of the actual known volume. This variation is due to the possible inaccuracy in measuring the column inside diameter for the known volume and due to planimeter error in the flowrate transient area. However when helium perturbation flow was added to argon the volume of the two voids was in close agreement.

The calculation in Figure 8.9.a when the perturbation flow is added to the system, gives similar results to those in Figure 8.9 b when the perturbation flow is removed. The area under the void, the offset and the residence time are the same in both graphs. From the graph in Figure 8.9.a the offset is  $8.9 \text{ cm}^3$  so therefore from planimeter reading :

$$\begin{aligned}
 \text{Actual area under the void peak} &= \text{Average planimeter reading} \times 15 \text{ cm}^2 \\
 &= 0.981 \times 15 \text{ cm}^2 \\
 &= 14.72 \text{ cm}^2 \\
 \text{Thus residence time (cm)} &= \frac{14.72 \text{ cm}^2}{8.9 \text{ cm}} \\
 &= 16.54 \text{ cm.}
 \end{aligned}$$

add

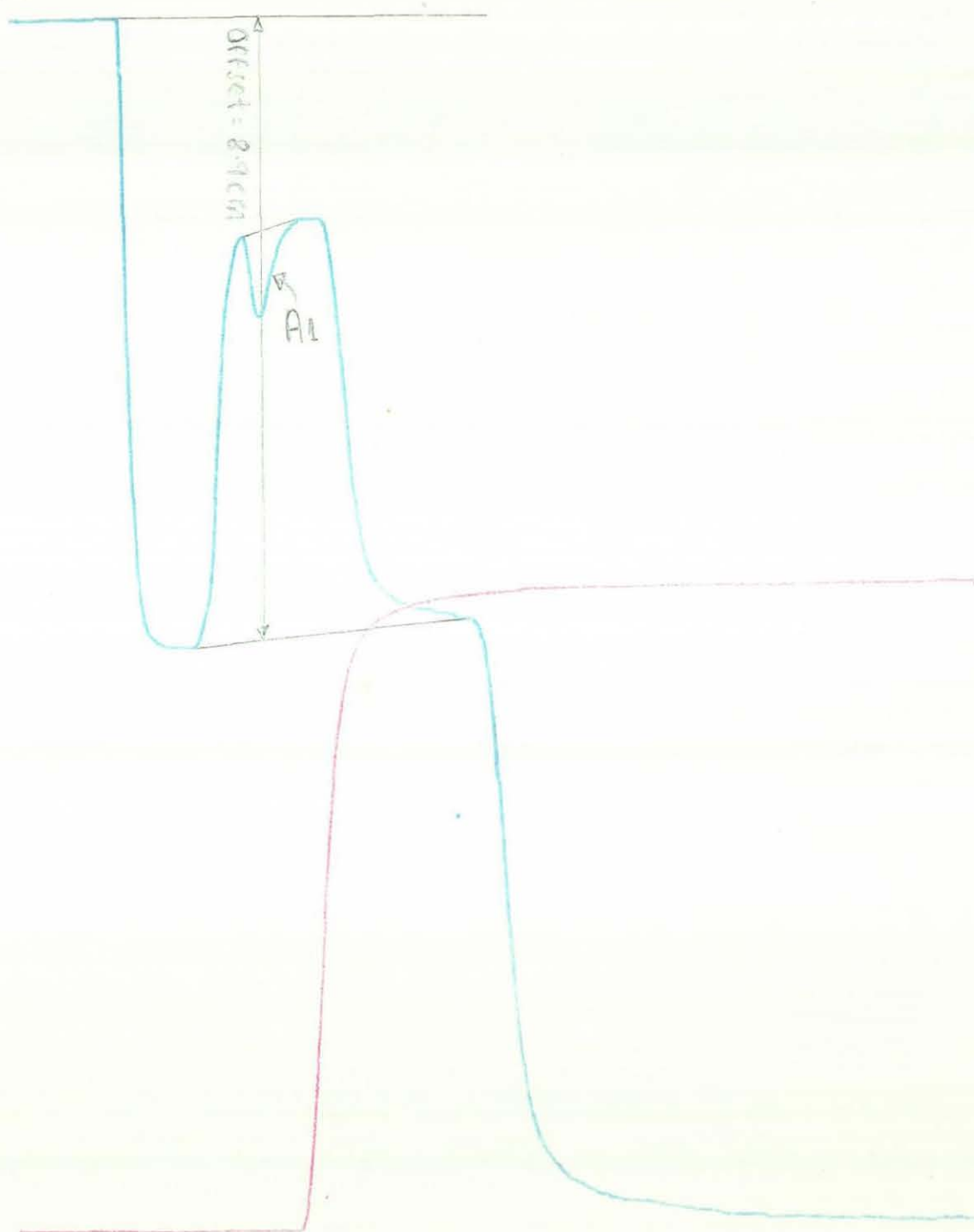


Figure 8.9.a Helium perturbation gas flow added to argon carrier gas



Figure 8.9.b Helium perturbation is removed from the system



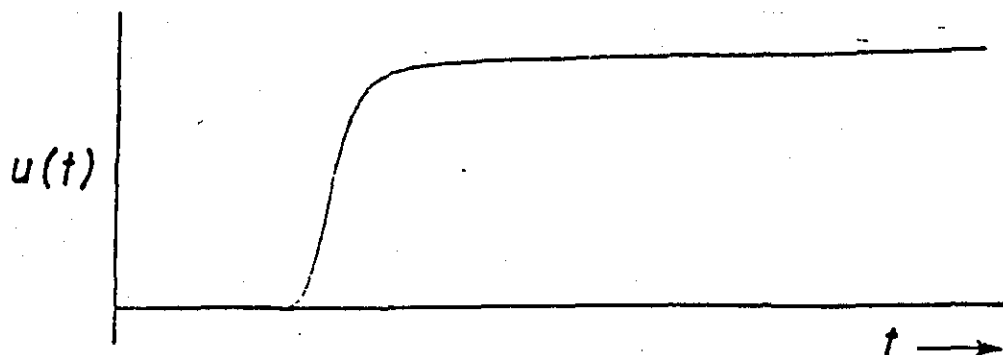
But we need to convert this distance to unit time and this requires a knowledge of the chart speed which is 20 mm/min.

$$\begin{aligned}\text{flow residence time} &= \frac{1.654}{2\text{cm/min}} \\ &= 0.827 \text{ mm}\end{aligned}$$

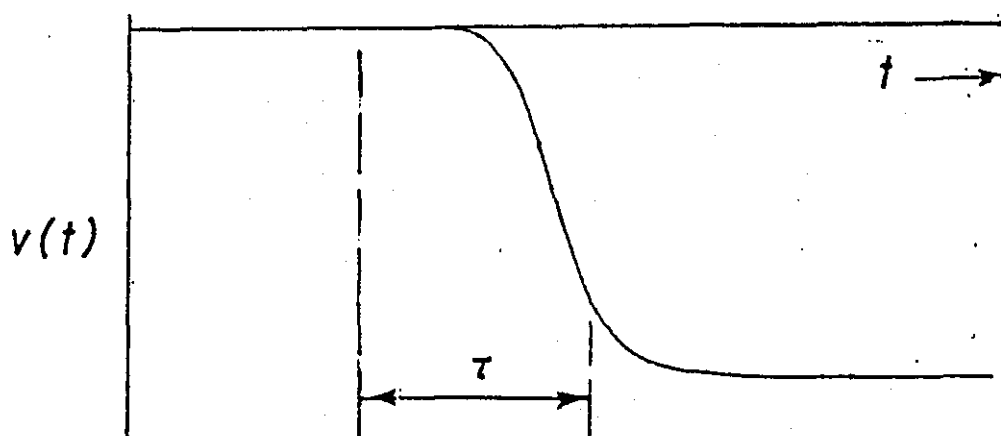
$$\begin{aligned}\text{flow residence time} &= 60 \times 0.827 \\ \text{for the whole column} \\ &= 50 \text{ s.}\end{aligned}$$

It is normal practice to ensure a constant baseline during the experiment. However, as the transient moves through the differential flowmeter, the baseline is changed due to the viscosity effect. This problem can be eliminated by inserting delay lines of sufficient length between the column and the flow-meter. This will result in a delay of the viscosity step such that the viscosity effect will not affect the flowrate transient as the perturbation flow moves through the column.

The theory derived below by Buffham [37] outlines the formula to find the volume of a void. Let  $u(t)$  be the flowrate deviation when the front leaves the packed section and enter the void i.e. adsorptive

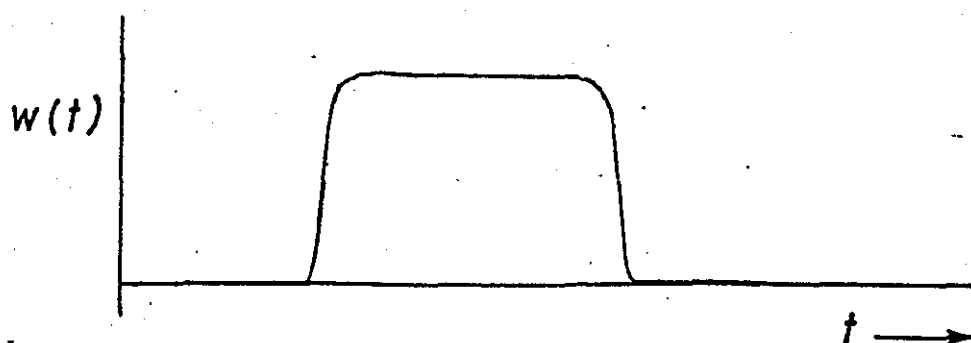


let  $v(t)$  be the flowrate deviation when the front enters the packed section downstream from void.



For a big void the effect is  $W(t)$ :

$$W(t) = u(t) + V(t) \quad (8.1)$$



If there is no mixing (spreading) in the void

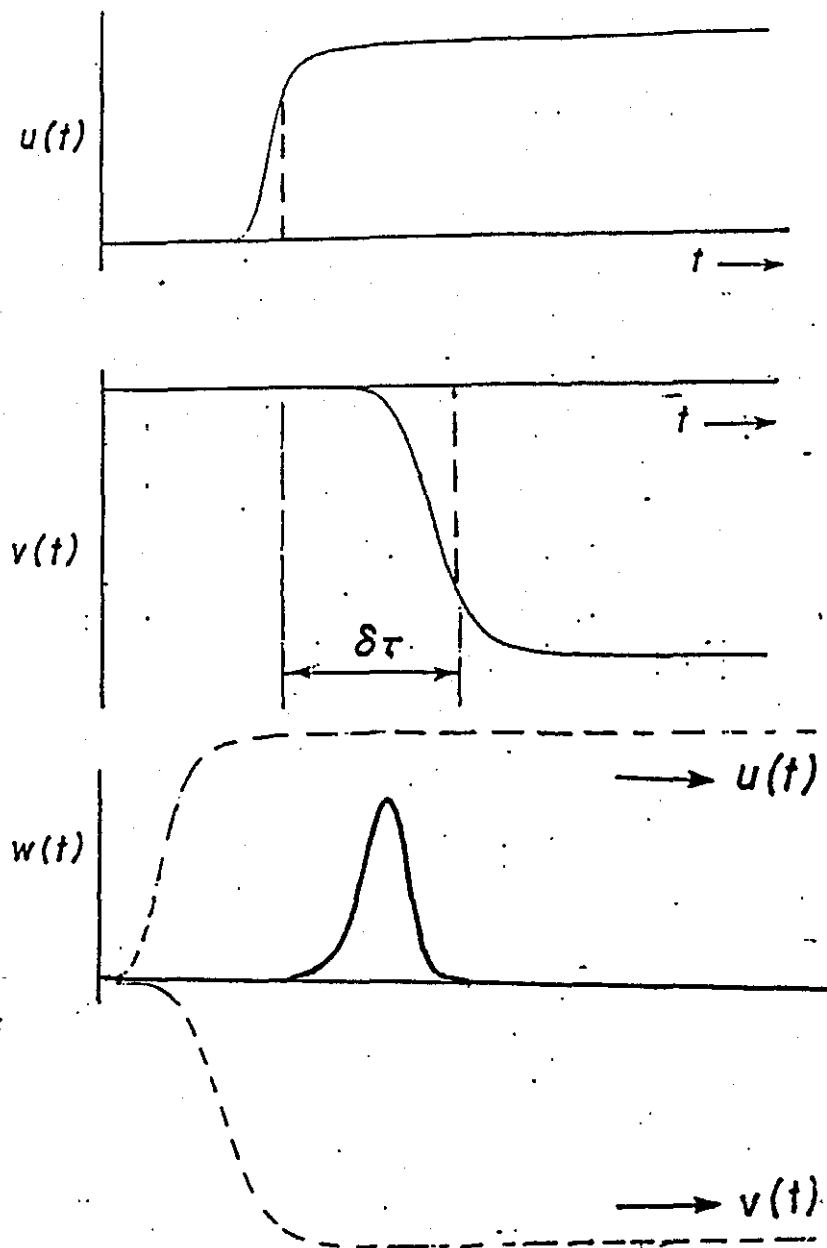
$$V(t) = -u(t - \tau) \quad \text{where:} \quad (8.2)$$

$\tau$  is the transit time across void

combining equations (8.1) and (8.2), eliminating  $v(t)$ , gives:

$$\text{so } W(t) = u(t) - u(t - \tau) \quad (8.3)$$

For a small void zone, let the transit time be  $\tau$ .



Now

$$w(t) = u(t) - u(t - \delta\tau) \quad (8.4)$$

$$w(t) = \frac{d}{dt}(t)[u(t)]\delta\tau \quad (8.5)$$



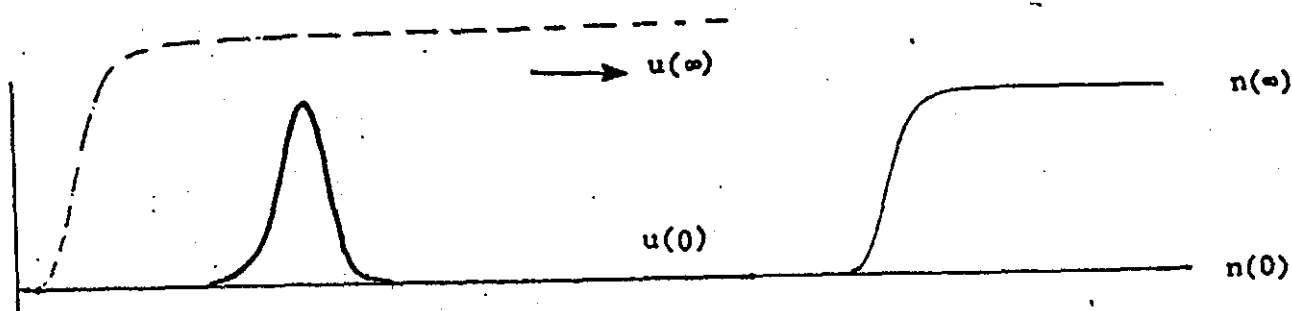
Integrate from time 0 to some arbitrary  $\infty$ .

$$\int_0^{\infty} W(t)dt = \delta\tau \int_0^{\infty} \frac{d}{dt}[u(t)]dt \quad (8.6)$$

$$= \delta\tau[u(\infty) - u(0)] \quad (8.7)$$

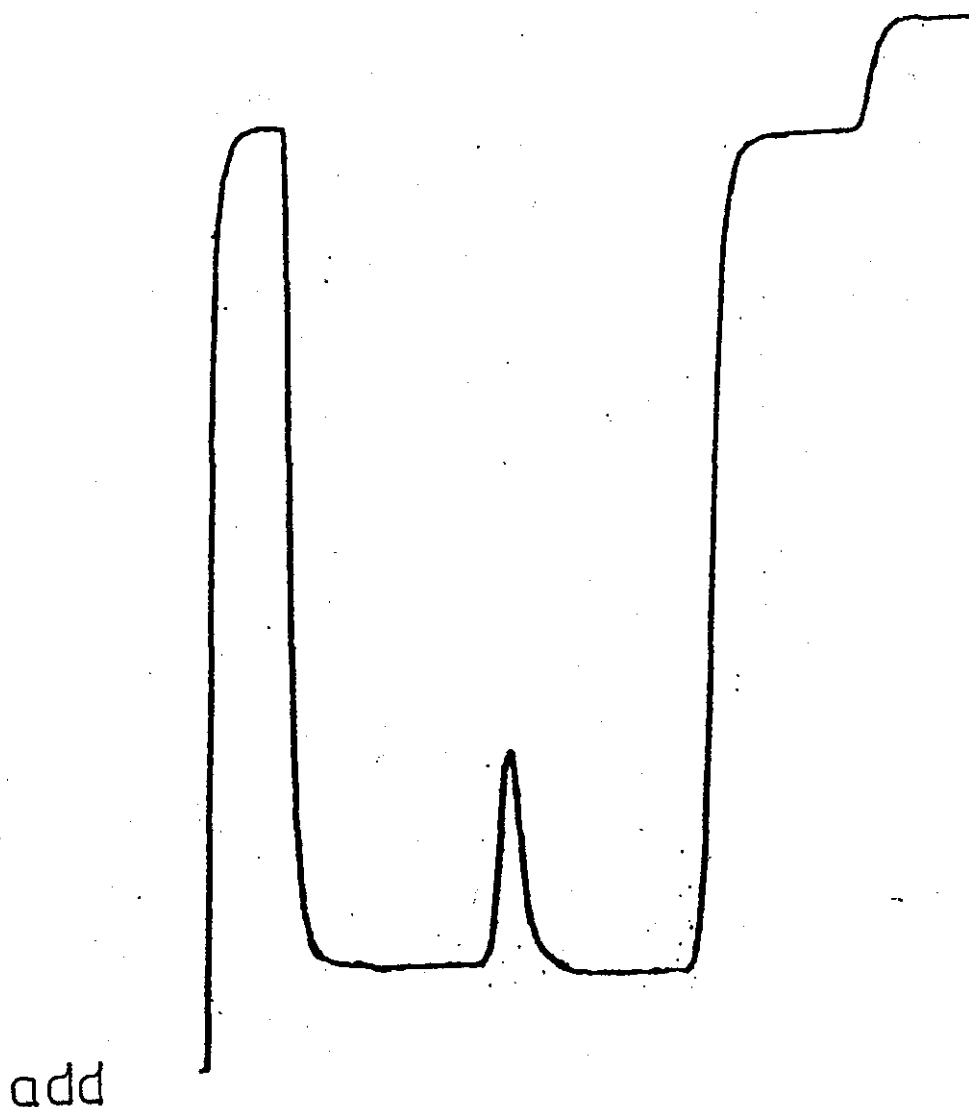
$$\delta\tau = \frac{\int_0^{\infty} W(t)dt}{[u(\infty) - u(0)]} \quad (8.8)$$

When the front eventually reaches end of column the chromatogram is:



$$\text{Offset: } N(\infty) - N(0) = u(\infty) - u(0) \quad (8.9)$$

The addition of the extra flow (the perturbation flow) to the system causes the offset at the beginning of the system as shown below.



Therefore

$$\delta\tau = \frac{\text{Area}}{\text{Offset}} \quad (8.10)$$

Figure 8.9 shows the void areas which were found by using a planimeter. The arm was positioned so that it was perpendicular to the planimeter body and the planimeter recording was noted. This was the starting point. The circumference was then traced around slowly taking care to follow the line as closely as possible and then the final reading was taken. This procedure was repeated until the readings were sufficiently consistent. With practice closely result could be obtained.

Initial Starting Point	Final Reading	Difference
8.555	8.596	0.041
8.596	8.637	0.041
8.637	8.679	0.042

Table 1 the area under peak 1

Initial Starting Point	Final Reading	Difference
7.850	7.890	0.040
7.890	7.850	0.040
8.850	7.809	0.042

Table 2

The results in table 1 and 2 for addition and subtraction peaks are consistent. From the planimeter arm setting we know that:

$$\text{every 1 revolution} = 15 \text{ cm}^2$$

$$\text{therefore our area} = 15 \times 0.041$$

$$= 0.615 \text{ cm}^2$$

from table 2 the area under the peak is:

$$= 15 \times 0.040$$

$$= 0.6 \text{ cm}^2$$

To find volumetric gas flowrate at the void in the column.

$$P_c = (P_{G1} + P_{At}) - \frac{X}{L}(P_{G1} + P_{G2}) \quad (8.1)$$

where

$P_c$  = pressure at the void.

$P_{G1}$  = gas pressure at 1

$P_{G2}$  = gas pressure at 2

$P_{At}$  = atmospheric pressure

This equation was derived in Figure 8.10 assuming that the pressure varies linearly down the column, an assumption which is valid for small pressure drops. The void is at the column mid - point so :-

$$P_c = P_{G1} + P_{AT} - \frac{1}{2}(P_{G1} + P_{G2}) \quad (8.2)$$

Every 1 bar = 750 mm Hg

$P_{G1} = 0.042 \text{ bar} = 0.042 \times 750 = 31.5 \text{ mm Hg}$

$P_{G2} = 0.010 \text{ bar} = 0.01 \times 750 = 7.5 \text{ mm Hg}$

Subs.in eqns. 2

$$\begin{aligned} P_c &= 748.4 + 1/2 (31.5 + 7.5) \\ &= 767.9 \text{ mm Hg} \end{aligned}$$

$$\text{Now } F_c = \frac{T_c}{T_{AM}} \times \frac{P_{At}}{P_c}$$

Where  $F_m$  = Flow rate at  $T_M$  and  $P_{AT}$

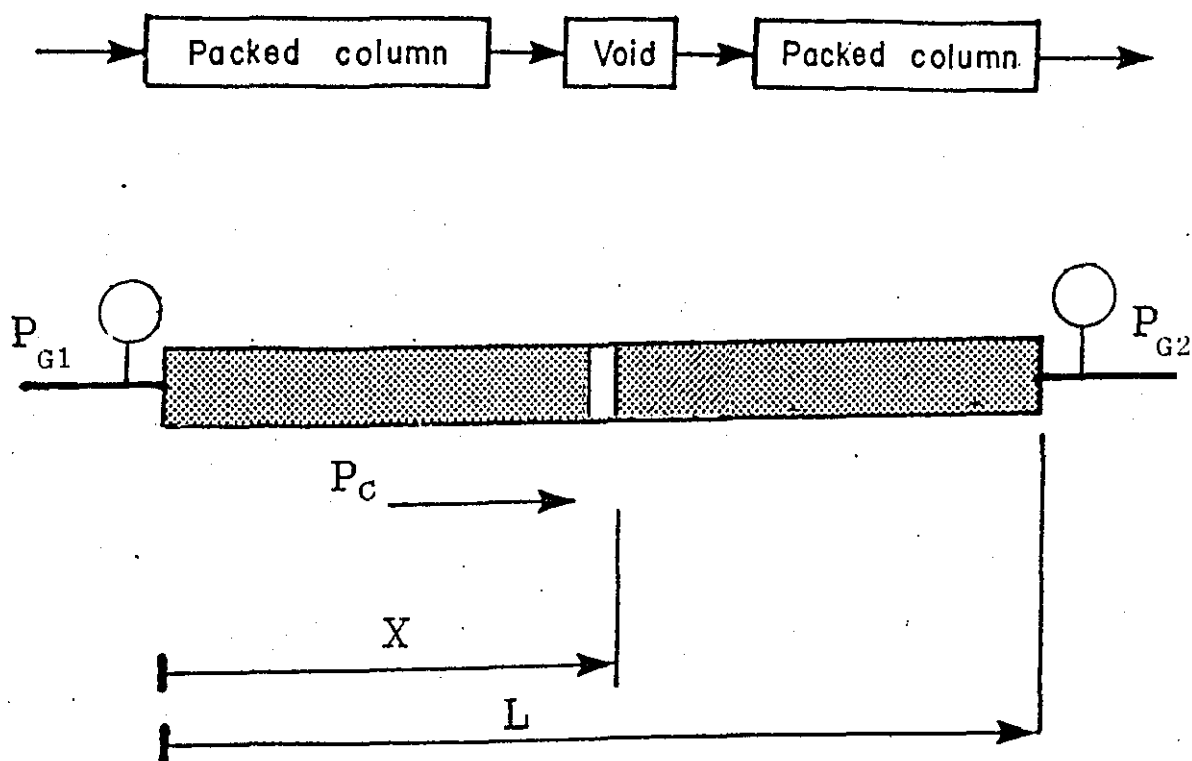
$T_c$  = Temperature must be absolute  $(C+273)^\circ$

$T_m$  = Room temperature

$$F_c = \frac{36}{2} \times \frac{60 + 273}{24 + 273} \times \frac{748.4}{767.9}$$

= 19.66 ml/min. Divide this by 60

$F_c = 0.3278 \text{ ml/sec.}$



$$P_C = (P_{G1} + P_{AT}) - X/L (P_{G1} + P_{G2})$$

$$\text{If } X = 1/2$$

$$P_C = (P_{G1} + P_{AT}) - 1/2 (P_{G1} + P_{G2})$$

$$= P_{AT} + 1/2 (P_{G1} + P_{G2})$$

$$P_C = P_{AT} + 1/2 (P_{G1} + P_{G2})$$

Figure 8.10 showing the derivation of the formula for the column pressure at the void.

$$\begin{aligned}
 \text{Void area under peak 1} &= 0.041 \times \text{planimeter no. of revol.} \\
 &= 0.041 \times 15 \\
 &= 0.615 \text{ cm}^2
 \end{aligned}$$

$$\text{offset} = 6 \text{ cm}$$

therefore

$$\frac{\text{Area (cm}^2\text{)}}{\text{offset (cm)}} = \text{cm} = \text{time(t)}$$

$$\frac{0.615 \text{ cm}^2}{6 \text{ cm}} = 0.1025 \text{ cm}$$

$$\text{but chart speed} = 2 \text{ cm/min.}$$

$$2 \text{ cm} = 1 \text{ min.}$$

$$\therefore \text{time} = \frac{0.1023}{2} \times 1 \text{ min}$$

$$= 0.051 \text{ min.}$$

$$= 0.051 \times 60 = 3.1 \text{ seconds}$$

$$F_c \times \text{time} = \text{void volume}$$

$$F_c \times \text{time} = 0.3278 \times 3.1 = 1 \text{ ml} = 1 \text{ cc} = 1 \text{ cm}^3$$

$$\text{Void area under peak 2}$$

$$= 0.040 \times 15$$

$$= 0.6 \text{ cm}^2$$

$$\text{Void volume} = \frac{0.6}{0.615} \times 1$$

$$= 0.975 \text{ ml}$$

$$\text{The cross-sectional area of the glass column was } 0.1257 \text{ cm}^2$$

therefore the length of the void

$$= \frac{0.975 \text{ ml}}{0.1257 \text{ cm}^2}$$

$$= 7.8 \text{ cm}$$

The actual length of the void was 6 cms and so the adsorption theory gives an estimate of the length of the void within about 10%.

### 8.3 Experiment Configuration Three

This experiment was carried out without the upstream delay lines. By removing the delay line, the resulting graphs will give actual values of composition retention time. The composition retention time ( $\tau_x$ ) can be found from the katharometer trace as shown in Figure 8.9 and Figure 8.10. However, it can also be found from the flowrate trace when operating the system without the upstream delay lines. The reading on the chart-recorder for the katharometer gives the starting point, offset and distance.

From the graph the area can be determined ( $\text{cm}^2$ ). Dividing this area by the offset(cm) gives a distance which corresponds to the residence time. Knowing the chart-recorder speed, this distance can be converted to a time. If the front is sharp, the area can be approximated to a trapezium, therefore

$$\text{distance to mid-point (cm)} = \frac{\text{trapezium area (cm}^2\text{)}}{\text{offset (cm)}}$$

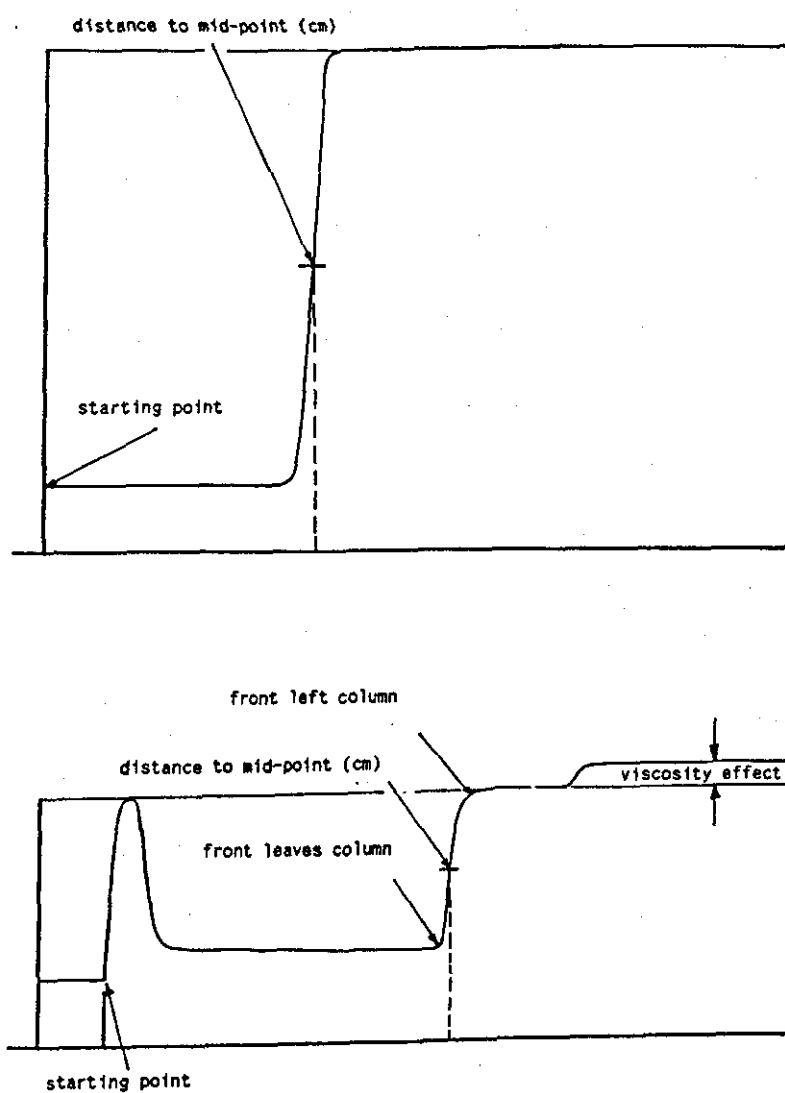
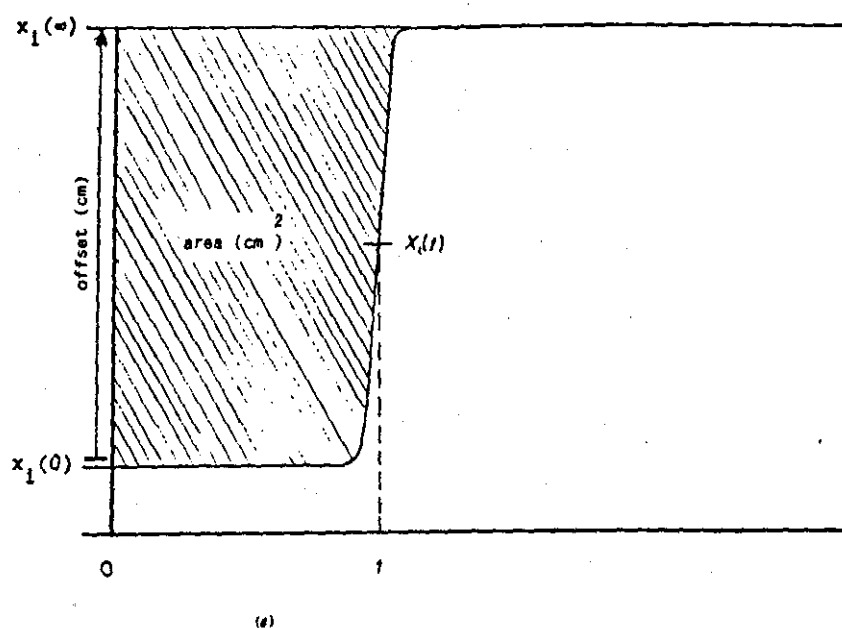
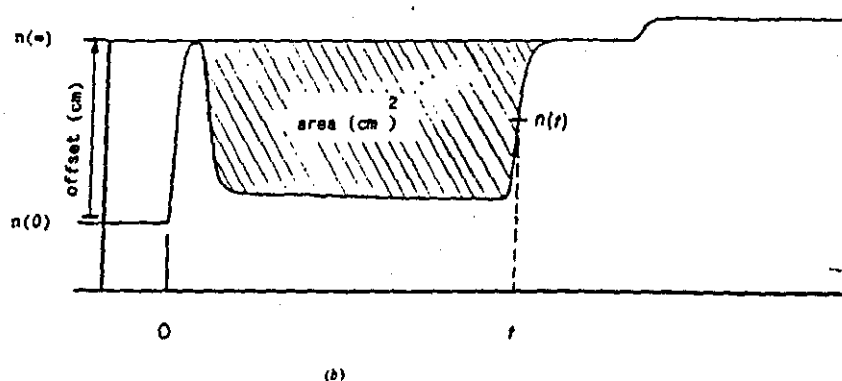


Figure 8.9 A typical gas chromatogram of nitrogen perturbation flow added to helium carrier gas. It also shows the various stages of a front passing through the system.





$$\text{Composition Residence Time (Tx)} = \int_0^{\infty} \frac{X_i(\infty) - X_i(t)}{X_i(\infty) - X_i(0)} dt$$



$$\text{Flowrate Residence Time (Tp)} = \int_0^{\infty} \frac{n(\infty) - n(t)}{n(\infty) - n(0)} dt$$

Figure 8.10 Perturbation variables (a) for the composition transient and (b) for the total molar outflow rate.

From Figure 8.10.a the planimeter measurement of the above area is 1.1805 and every one revolution of the planimeter is equal to 15 cm<sup>3</sup>, so therefore area:

$$\text{Area} = 1.1805 \times 15$$

$$= 17.70 \text{ cm}^2$$

$$\text{Offset} = 7.2 \text{ cm}$$

converting into time: 1 cm = 1 min.

$$\tau_x = \frac{17.70}{7.2} = 2.46 \text{ cm} = 2.46 \text{ min}$$

Converting into sec: 2.46 x 60 = 147.5 s.

From the blue trace Figure 8.10 b the distance measured from the start to halfway up the front leaving the column was calculated manually and it was found to be 2.48cm. By comparing the two results above taken from the katharometer trace and the perturbation flow, the composition retention time ( $\tau_x$ ) is the same. Similar calculations were also carried out in Figure 8.11 to Figure 8.13.

Figure 8.11 shows the results of adding a nitrogen trace to helium bulk flow. The distance obtained by approximating the red trace to a trapezium is 78.5 mm. This is the normal way of measuring composition retention time. By the new method of using the blue (flow-rate) trace, a distance of 79 mm was obtained. In this case, the method gives a very good agreement with the standard method. In Figure 8.12 the two methods were compared for adding a helium trace to argon bulk - flow. The distance by approximating the red trace to a trapezium is 19 mm. This is the normal way to obtain  $\tau_x$ . By the new method of using the blue trace, a distance of 19 mm was obtained. Thus this the new method gives similar results to the standard method.

In Figure 8.13 argon perturbation flow was added to a helium carrier gas. The calculation procedure was repeated and by approximating the red trace to a trapezium the distance 98 mm. By the new method of using the blue trace, a distance of 97 mm was obtained. So, for adding helium perturbation flow to argon this new method also gives a good

agreement when compared to the standard method. So, therefore the results obtained from the new method are similar to the conventional method and it can be concluded that the composition retention time  $\tau_x$  can be obtained from the flowrate trace as well as from the katharometer trace.

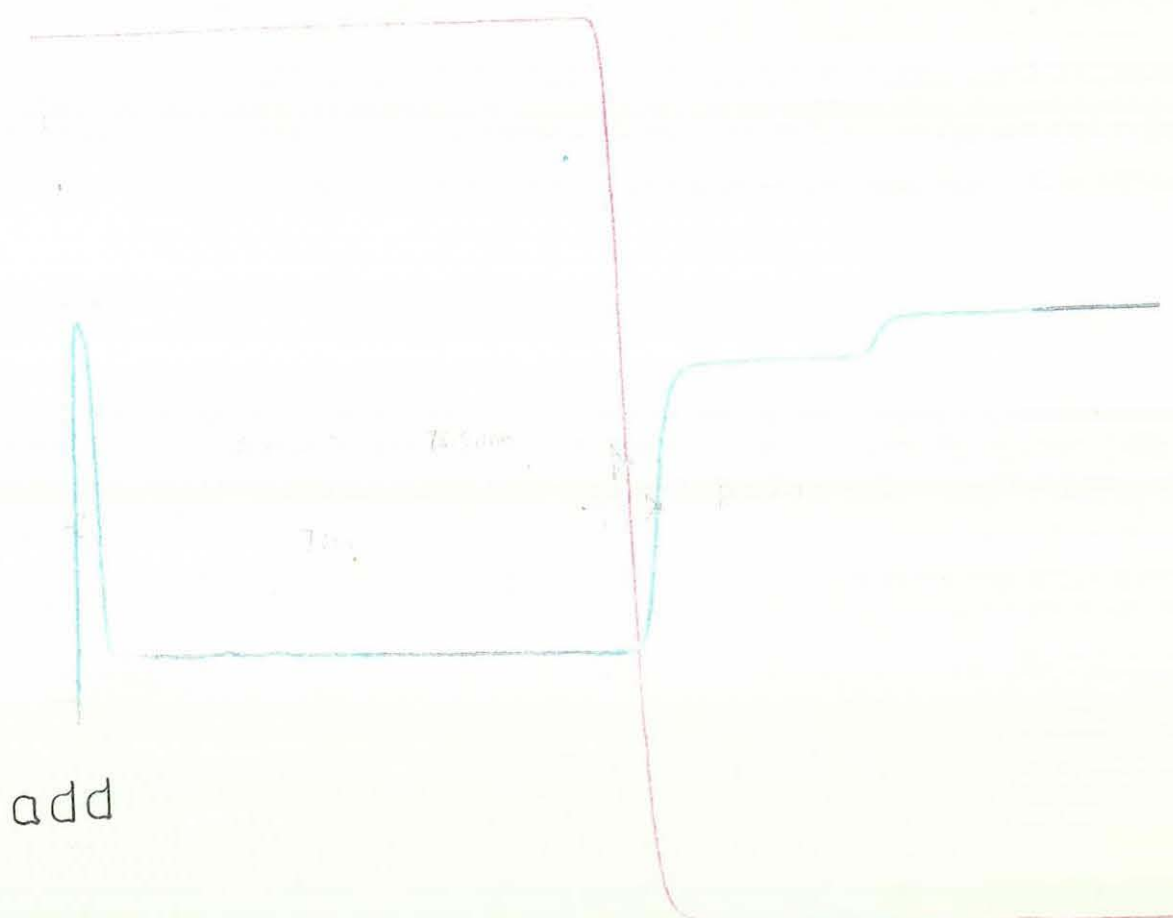


Figure 8.11 Chromatogram resulting from a 0.45% nitrogen perturbation flow added to helium carrier gas without up-stream delay lines.

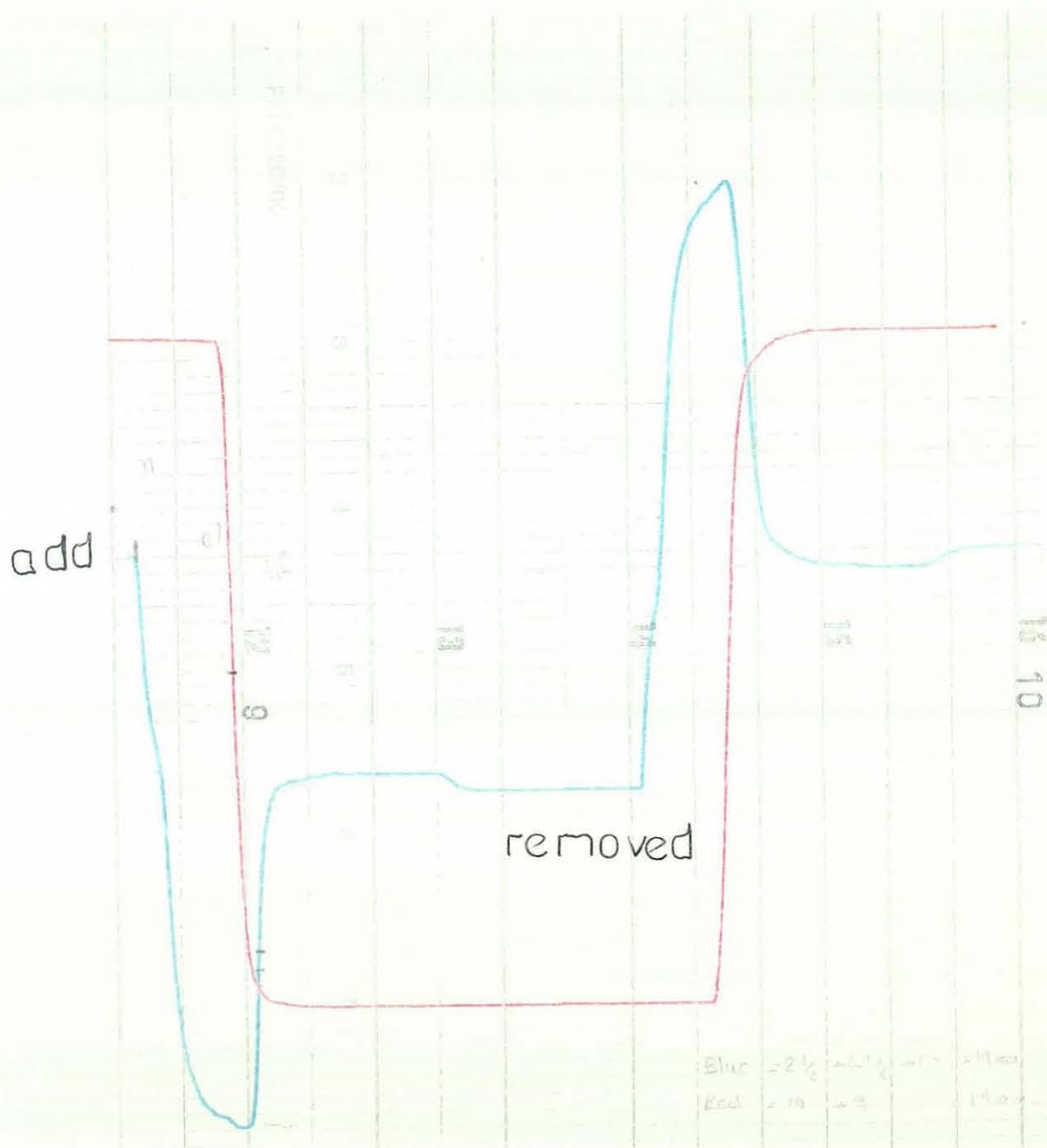


Figure 8.12 Chromatogram of a 0.5% nitrogen perturbation flow added to helium carrier gas. Without up stream delay-line.

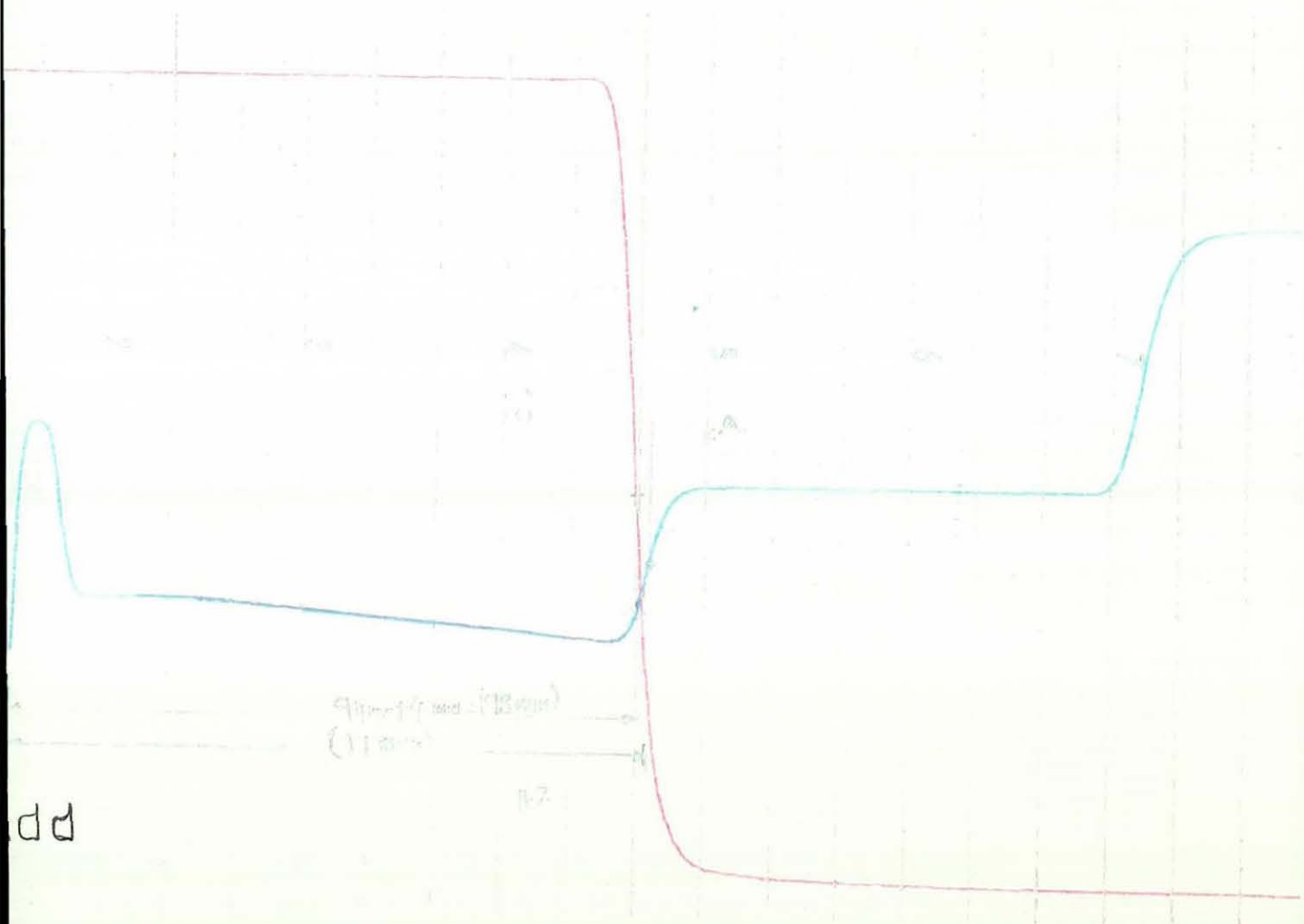


Figure 8.13 chromatogram resulting from a 0.5% argon perturbation flow added to helium carrier gas. Without up stream delay line.

## 8.4 Flowrate Residence Time Calculations ( $\tau_p$ )

In this section calculations are carried out to obtain the flow-rate retention time ( $\tau_p$ ). This can be found, with or without the upstream delay line, from the flowrate (blue) trace only. The basic formula to calculate the retention time  $\tau_p$  is to divide the area by the respective offset.

Integrating between time zero and time infinity.

$$\begin{aligned}\tau_p &= \int_0^{\infty} \frac{n(\infty) - n(t)}{n(\infty) - n(0)} dt \\ &= \frac{1}{n(\infty) - n(0)} \int_0^{\infty} n(\infty) - n(t) dt\end{aligned}$$

The area can be found in various way: Firstly by measuring it with the planimeter or by dividing it into squares and shapes. Figure 8.13 shows the area when nitrogen perturbation flow gas is added to helium bulkflow; measuring the area with the planimeter gives 11.8 cm<sup>2</sup>. Therefore

$$\begin{aligned}\tau_p &= \frac{11.8 \text{ cm}^2}{4.8 \text{ cm}} \\ &= 2.458 \text{ cm} \quad \text{every 1 cm} = 1 \text{ min.} \\ & \quad 2.0458 \text{ cm} = 2.0458 \text{ min.} \\ &= 2.0458 \times 60 \\ &= 147 \text{ seconds.}\end{aligned}$$

Thus the values of  $\tau_x$  and  $\tau_p$  can be found from the flow rate (blue) trace only. If we data log the flow rate signal and produce appropriate software then  $\tau_x$  and  $\tau_p$  can be calculated easily. For computer data logging and computer programming refer to appendix B.

## CHAPTER NINE

CONCLUSION AND RECOMMENDATIONS

---

## 9.1 Conclusion

- (1) The objective of this work has been to develop an understanding of the detection of voids in packed chromatographic columns using small flows of a perturbation gas. The results obtained during the course of the project show that it is possible to detect the size of the voids in packed chromatographic columns using a small flow (0.5% of the main flow) of perturbation gas.

Using one column and a high chart recorder sensitivity, flow rate changes were observed, which are attributable to the gas front running past the void in the system. The area under the peak is larger when a highly sorbed carrier gas (nitrogen) is used with a weakly sorbed perturbation gas (helium). The peak area is the net result of an amount of perturbation gas desorbing and the carrier gas resorbed.

- (2) The sensitivity of the apparatus could be increased by reducing the sensitivity of the chromatographic columns to the effects of thermal noise by using a truly isothermal oven. From the preliminary results, thermal noise was seen to be a potential problem, and so modifications to the apparatus were made to reduce this noise. When the oven is on, the flowrate record is susceptible to thermal fluctuations reducing the smoothness of the flowrate record and making void area determination, and retention time measurement difficult.

Preliminary investigations showed that operating the oven at low temperature would reduce the fan noise level. Also running the apparatus at (room temperature) would reduce the system fluctuation.

Random surges in atmospheric pressure were observed to cause rapid fluctuations in the gas flow meter. This is because of volume distributions imbalances between the two flow channels of the chromatograph so that an external pressure fluctuation causes a pressure surge to be propagated through the two sides of the chromatograph at different rates and thus affect the DPT at different times. However the employment of the re-designed BPR and using one side of the system only reduced the system noise level to such an extent that the baseline noise level for nitrogen, helium and argon became indistinguishable; previously the noise level for more adsorbed nitrogen was very high.

- (3) The purpose of the perturbation trace mixing device is to enable the perturbation flow to be exactly cut off and sharply added. Previously, there was always a "dead-volume". After the perturbation flow was switched off perturbation gas would continue to diffuse out of the dead volume. The new invention of a trace mixing device has solved the dead volume problem. This has enabled all perturbation flow addition and removal runs to be mirror images of each other.
- (4) Delay lines have been extremely useful in this research. Which were situated in two locations of the apparatus: one was located between the TCD and the DPT; (down stream). The main purpose of this location is to allow direct determination of the flowrate retention time. The second location was between the column and the perturbation trace mixing device (upstream) of the column. Originally, when the the perturbation flow was switched from one column to the other, only an upstream delay line was employed. This was done to separate both flowrate and composition transients for both adding and removing the perturbation flow, although the delayed transients were distorted with slight differences in sharpness, and shape. Later on, a further development to the apparatus allowed a perturbation flow gas to be either added to or removed from one side of the system, the same system behaviour was noted. These findings were confirmed experimentally. The downstream delay line enabled the flow rate to be measured at constant composition. Only when all of the composition front has finally left the column does a composition change occur at the capillary flow meter and cause the viscosity response.



- (5) The determination of equilibrium isotherms for single or multi-component (binary isotherm) depend upon the system pressure and temperature, and so it is important that the gas and chromatographic column are always at constant temperature.

Adsorption equilibrium is commonly expressed in terms of an adsorption isotherm. The isotherm is the relationship between the amount of gas adsorbed on an adsorbent and the amount in the gas phase. For a binary system this basically involves adding the required mixture to the adsorbent and waiting for equilibrium to be attained before performing a molar balance. Because the adsorbed gas phase composition is unknown, an extra measurement is required to perform the motor balance. The gas phase composition determined by gas chromatography is not enough.

However, in the experiments reported here only pure bulk gas flow were used and so it was only possible to obtain the isotherm gradient at the composition end points. The experiments showed the method is simple and quick, making it ideal for preliminary adsorbent screening.

- (6) The distribution of pressure drop in the system was a possible cause of error. The size of the peak produced by both adsorption and desorption of the font passes the void in the column could be affected by a high pressure drop in the column. The solution was to have a uniformly packed column with a low pressure drop which would have little effect on the peak area. The calculation of the peak area can be approximated by measuring of the peak height and the peak width at half the height, or by constructing tangents to the peak at the inflection points and using the area of the constructed triangle to represent the peak area. More accurate areas can be obtained by a method known as cutting and weighing where each peak area is cut out of the chart paper and weighed. This method relies on the paper being homogeneous and it also destroys the chromatogram. Apart from being a most tedious method, the area can be determined by counting of squares. However, a planimetry technique was used here where a perimeter of the peak area is traced using an instrument known as a planimeter. This method were proven to be suitable for measurement of small areas.

- (7) Operating the system without upstram delay lines, it has been shown by experiment that from the perturbation flow trace (chart-recorder print out) the values of composition retention time ( $\tau_x$ ) and pressure retention time ( $\tau_p$ ) can be found respectively. Previously, using a standard method, it was thought that it is necessary to use the composition transient to determine the values of composition retention time  $\tau_x$ . Using the new method proposed by Mason and Buffham [35] enables both  $\tau_p$  and  $\tau_x$  to be determined using only the flowrate transient.

## 9.2 Recommendations for further work

- (1) Further work, based on the preliminary work shown in Appendix I, linking the computer with the chart recorder to achieve real time data processing.
- (2) Further work on the computer programmes. These programmes need to be linked together to perform as a single operation.
- (3) Further work on the programme in order to scan the data and perform calculation of  $\tau_p$  and  $\tau_x$ .
- (4) Recommendations to integrate the spreadsheet Quattro to the main program. This will enable the operator to import data to the Quattro programme to review it and immediately display the graph where calculation would be carried out. The computer calculation will produce faster and more accurate results.

## CHAPTER TEN

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## APPENDIX I

### Graphics Analysis & Temperature Measurement

## APPENDICES

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## APPENDIX I

### - Graphics Analysis and Temperature Measurement

#### I.1 Introduction

In Chapter 8, the theoretical procedure for calculating  $T_x$  and  $T_p$  was demonstrated. This method of calculation has proven to be simpler and easier to follow and it is intended to demonstrate the simplicity of this method using a computer. The industrial computer source ICS card has been used for data logging signals and converting them into digital signals. This digitised signal (data) is received by the computer to be processed in a real time operation at 2,500 samples per second with a high level of accuracy.

The addition of the ICS [38] card Mini - 16 model, to the IBM 30-286 computer, where all data is received has shown the system to be highly accurate in processing data and faster than similar function programme used by Meacham in his research. The IBM computer receives the output signal from the DPT, through the termination box (T.35) and the card which is plugged into one of the peripheral slots inside the computer.

However further improvements were carried out to optimise the detector, the separation, and the reduction of pressure drop and signal (noise). In this application of gas chromatography, the programming languages offer faster access, and precision. The ICS card also has an integrating converter to reduce noise level during the processing time.

#### I.2 System Hardware

The data logging hardware consists of three major elements:-

- (1) IBM computer
- (2) Analog card
- (3) Termination box.



### I.2.1 IBM Computer

An IBM Computer PC, 30-286 computer with 256K RAM memory was used to analyse data and display the perturbation flow changes. Since the computer cannot represent an analog value, an analog-to-digital converter was used to change the analog signal into a series of number bits. The converter is 16-bit resolution, which means it will resolve 1 count out of 2 raised to the 16th power (65536). The resolution of the analog-to-digital converter is 0.0015%. The DPT output signal is also received by a conventional chart recorder with two channels, which can be read simultaneously.

### I.2.2 Analog Card

The analog / digital MINI-16 card shown in Figure I.1 is employed to simplify data acquisition and control the data received through the termination box T.35. This data logging card is also used to measure the temperatures of the columns and the katharometer cell. The device measures both temperature by simply selecting the appropriate menu. It is also used to receive differential analog inputs. This device proved to be ideal for this research environment.

### I.2.3 The Termination Box

The termination box T.35 shown in Figure I.2 is connected to the analog card inside the computer with one cable. It plugs into the edge of the card at the back of the computer. All input and output signal connections are made inside the terminal panel. It also has a cold junction to compensate for RTD's thermo-couples and a set of resistors for signal conditioning.

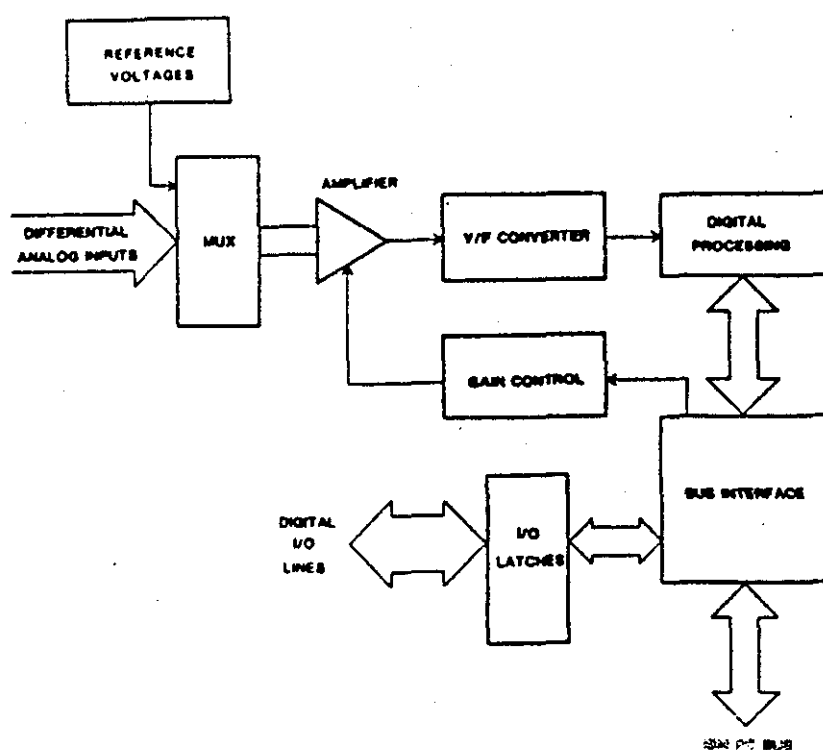
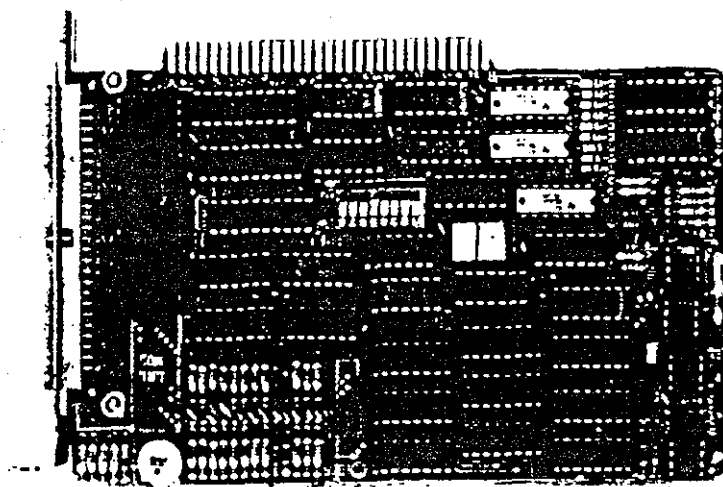


Figure I.1 Show the MINI-16 analog/digital card

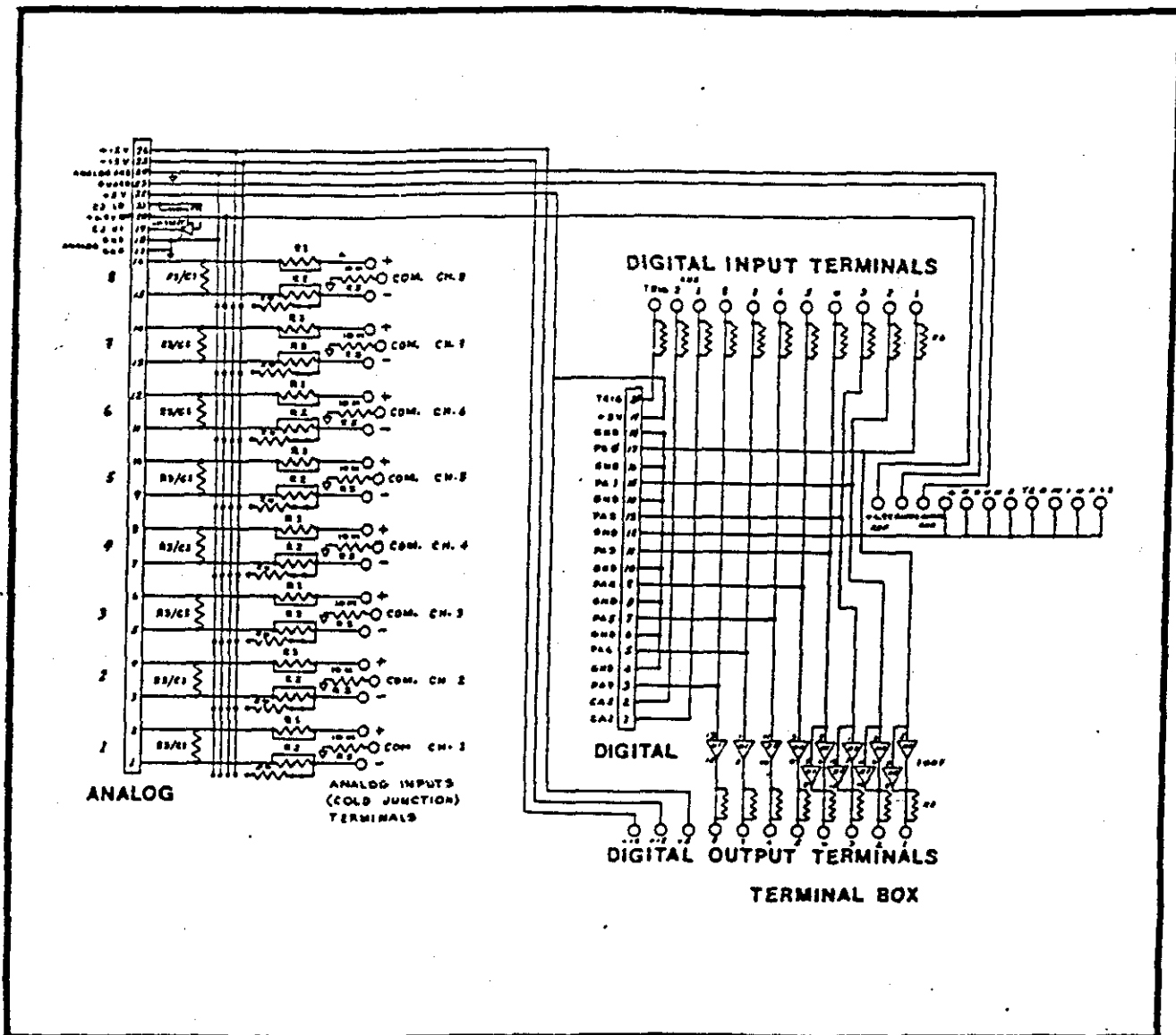


Figure I.2 The termination Box T.35.

### I.3 Resistance Temperature Detectors (RTD's) Installation

Two four-wire RTDs were used to sense both the temperature of the Pye 104 Oven and the katharometer cell. These platinum resistance probes measure in the temperature range ( $-50^{\circ}\text{C}$  to  $+250^{\circ}\text{C}$ ) with a time constant less than 2 sec. These probes are rugged, accurate and can detect temperature with precision. It also has a special feature in which it does not rely on the signal wires to be of equal resistance.

Figure I.3 shows the connections of the 4-wire RTD's and Figure I.4 shows the resistance/temperature relationship and tolerance for these B.S. 1904 Grade II (DIN 43760) probes. The source of power is 6.9 volts, and the resistor  $R_3 = 49.9\text{K ohms}$ . This resistor should be precise and stable enough to keep the voltage drop across the RTD's below 50 mV in order to keep heating less than 50 microwatts and variation of temperature errors negligible.

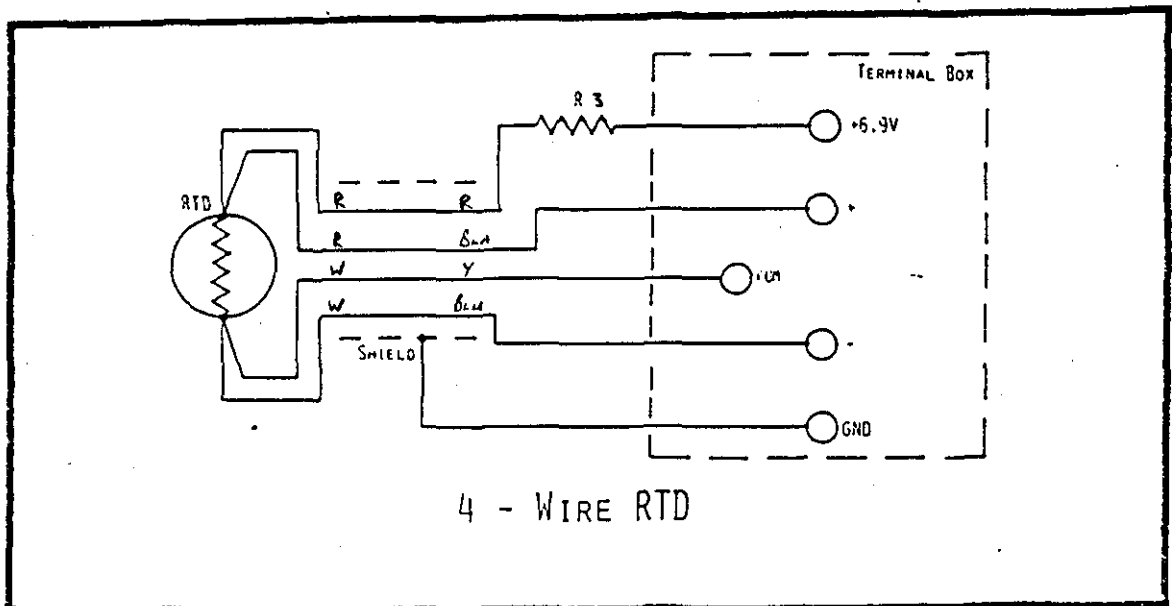


Figure I.3 shows the 4-wire RTD's connections.

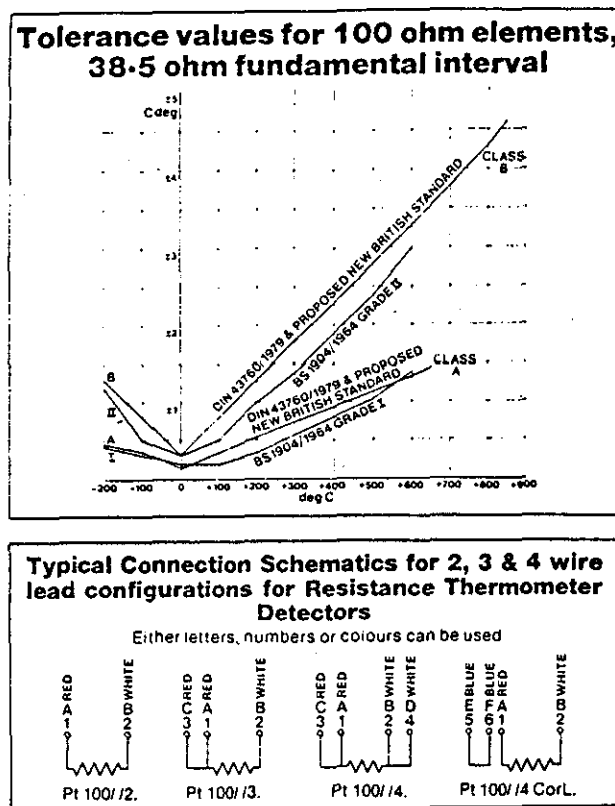


Figure I.4 The resistance/temperature relationship

## I.4 System Software

The data logging ICS card software consists of the following program:

I.4.1	Main menu program	CHROMB. BAS
I.4.2	Data logging program	LOGDAT. BAS
I.4.3	Data storage program	DATASAVE. BAS
I.4.4	Data display program	ADISGRPH. BAS
I.4.5	Data processing	DATAPROC. BAS
I.4.6	Calibration program	AUTOBOOT. SET

These programs will be discussed in detail later in this chapter but first it is important to outline the overall improvement of this software.

## I.5 Software Program and Improvement

While using the MINI-16 card programs some areas of improvement became apparent. Some of these changes were required to adapt the system to our use and can be seen purely as development work rather than fundamental rewriting. The software provided by ICS needs development, modification, debugging, and rewriting some of the programs, to accomplish a fully working system. These area of work and development are:-

- (1) Modifying the main menu by adding extra features to simplify the access to the main program; as soon as the computer is switched on the user is faced with master menu giving the options to select the appropriate choice. The second menu gives the user the option of selecting the soft ware to drive the ICS card.
- (2) De-bugging and modifying the "ASETUP.BAS " by removing of the logging of the digital I/O status and modifying the program to store data. Further work was carried out to the analog inputs data in line 10030 of the ADISPLAY.BAS program to read data into array and convert it to the units specified in the set up mode.
- (3) Writing an analysis program, to scan data and display graph to performed calculation.
- (4) Explaining each program procedure and providing a flowchart, to simplify the program understanding.
- (5) Re-writing software program to perform calculation of Tx and Tp to accommodate the changes of the communications between programs.

Although the programs are written on a modular basis they successfully performed the task required. However these programs need to be linked together to perform within a single operation.

### I.4.1 - Main Menus

All main menu procedures have been written to permit the selection of a number of options. The options available within the master menu are as follows:-

- (1) Turbo Basic
- (2) Turbo pascal

By entering the selection number of the programme the main control menu will automatically chain the main programme to the choice required. To run the system, see below and Figure I.5 flowchart detail.

[illegible]

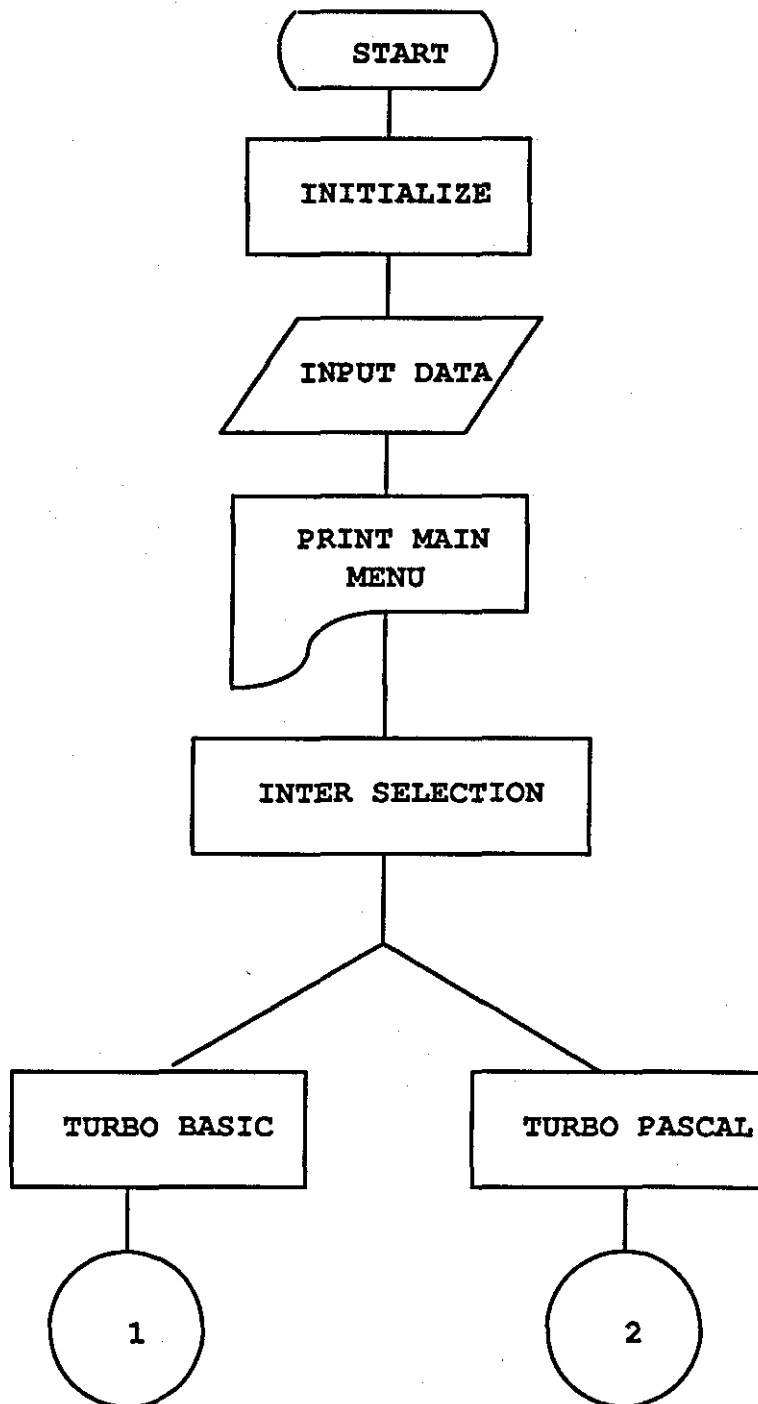


Figure I.5 Master menu condensed flow diagram



#### I.4.2 Data Logging Program

The data logging program is structured in such a way that data can be stored automatically without any intervention, simply by selecting the appropriate key. It is also possible for data to be logged manually with the option of logging the data on disc or printer. This information is entered before any data logging has been done. Data may also be logged to a different file. If the logging data is more frequent than every 10 seconds, the disc file will remain open until exit from the graphing mode. Then the computer permits the user to enter the data log title (see flowchart Figure I.6 and program listing Appendix II).

However, this program allows the user to log data on any disc driver or subdirectory and will not permit the user to leave the set-up menu unless a file name and time interval are entered, or if the user chooses to run off the data logging.

#### I.4.3 Data Storage Program

All data that is displayed on the screen can be dumped to the printer or disc. The data which is logged on disc is saved in a text file, compatible with the Quattro [39] spread sheet and the data analysis program. To analyse the data, simply import the file where data can be then reviewed and loaded into columns as in the Appendix II for data listing.

#### I.4.4 Data Display Program

This program displays a selection of numbers and letters of data display. It also show the last reading, and the present value of the analog inputs in a columnar display. The channel name and units are also displayed, the user can quickly read the exact values and confirm the units and location of an analogue input. In operation, the temperature of columns and katharometer cell is monitored for each runs, it also displays the difference between both RTD's channels and the current time. The display program allows the user to read data and display this data at full speed into the memory or log the data to disc when required. The resulting sample rate at which the channel will be read is 1.8 milli-seconds per channel.

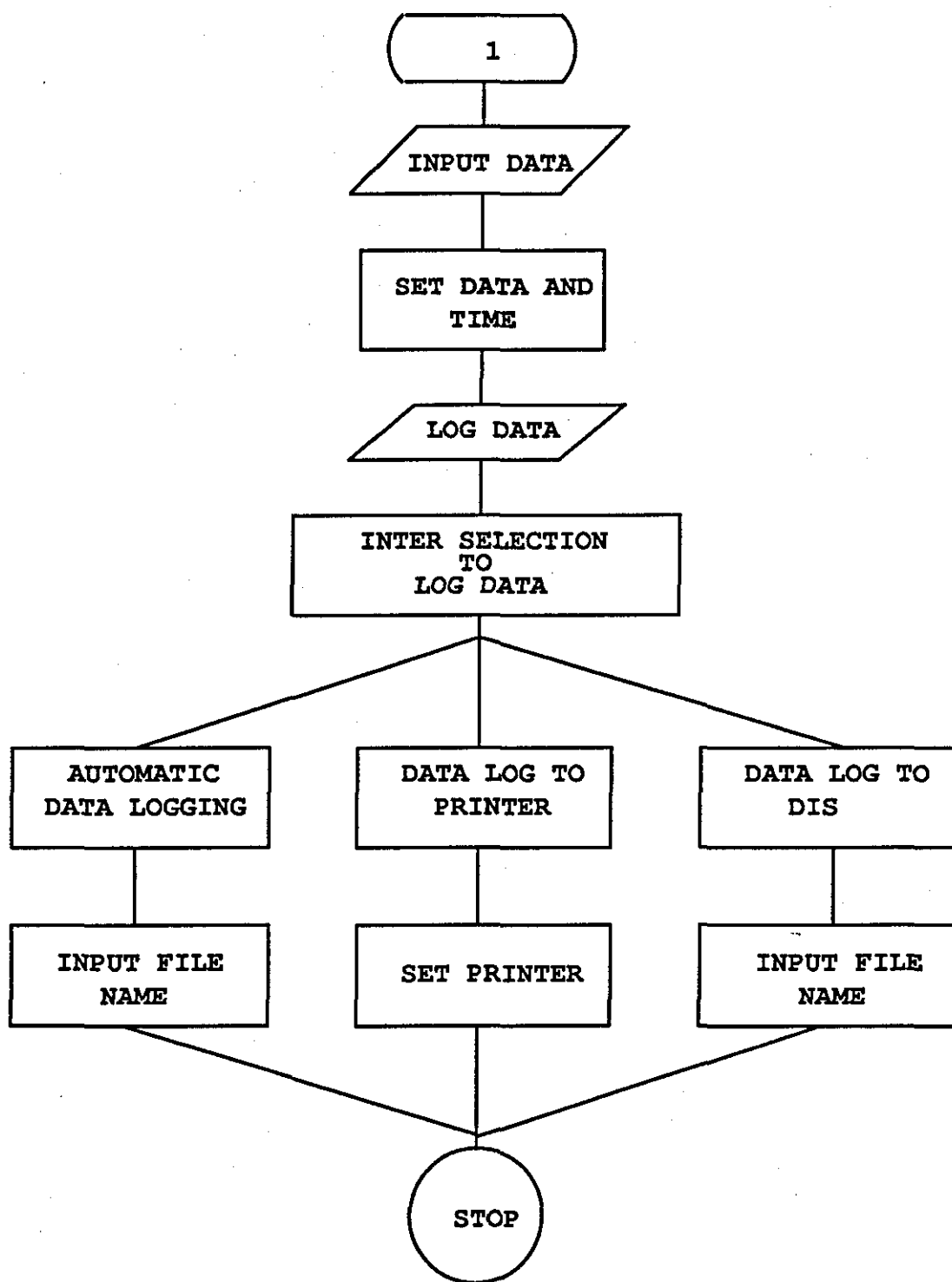


Figure I.6 Data logging programme flow diagram.

#### I.4.5 Data Processing Programs

The analysis programs is written to read data from a file then this data is displayed in the graphing mode, the resulting graph would be displayed on the screen in real time immediately upon entering the display mode.

AUG 05 1991 11:32:08 AM

Present analog inputs

Input Channel 1	22.1919	Degrees C
Input Channel 2	22.07541	Degrees C
Input Channel 3	-3.360445E-03	volts

Difference

Channel 1 - 2	.1164856	Degrees C
---------------	----------	-----------

Reset AUG 05 1991 11:31:52 AM

Maximum / Minimum

Input Channel 1	22.1919	22.17251	Degrees C
Input Channel 2	22.13047	22.07541	Degrees C
Input Channel 3	-3.359907E-03	-3.677516E-03	volts

Average / Number of samples

Input Channel 1	22.18	Degrees C	6
Input Channel 2	22.11	Degrees C	6
Input Channel 3	-3.51018E-03	volts	6

#### I.4.6 Calibration Program

This program (AUTOBOOT.SET) provides a unique technique to obtain a high stability set-up without any further adjustment for a very long period of time by using an adjustment potentiometer to calibrate the card. This adjustment is carried out by using the software which provides absolute zero drift in the calibration. The analog inputs are automatically recalibrated during operation by entering the calibration number on to the analog card to the program in the set up mode.

##### INPUT

CS = "n" + CHR\$(0)

The variable A%(x) must be dimensioned to have at least 8 elements: DIM A%(7) if the first element is A%(0). A%() is an integer array.

##### OUTPUT

All arrays = 0 if no driver installed.

A%(0) = total number of analog inputs installed

A%(1) = total number of analog outputs

A%(2) = total number of digital I/O's installed. = -1  
if FIND not run but driver installed.

A%(3) = 0

A%(4) = total number of analog output channels with  
valid calibration numbers

A%(5) = total number of analog input channels for which  
there are CCAL numbers

A%(6) = total number of analog input channels for which  
there are calibration numbers (ACAL, BCAL, and  
DCAL)

A%(7) = address of first board with a clock

A%(4) and A%(5) = -1 if no card installed or FIND not run.

100 ' Are calibration numbers correct?

200 DIM A%(7),B(7)

300 CS = "n" + CHR\$(0)

400 CALL AM1(A%(0),B(0),CS)

500 IF A%(0) > A%(5) OR A%(0) > A%(6) THEN PRINT  
"Calibration numbers are not correct":END

In gas chromatography data processing the amount of chart paper needed would be very large. In the case of a process requiring a recorder chart speed of 12 inches per minute, in a 24 hours period the recorder would use a quarter of a mile of chart paper. Several techniques have been developed [40] to simplify the record and economise on paper. The earliest method is the bar graph which is used to present peak height or the values of area under the peak. In the past the bar graph method was used almost exclusively for analogue presentation of peaks for data processing of gas chromatography. In another method used by R. Meacham, the output signal has been read out by a differential input interface CIL Model PCI 1281 linked via an RS232 port to the BBC Master 128 computer. The interface receives the output voltage from the DPT and this gives one of a number of 14 input channels which could be read simultaneously.

In this research the method used presents a simple, easily interpreted record of data using a reasonably small amount of paper. It does, however, require some degree of linkage to the computer. The ICS card is linked to the IBM computer to achieve fast, accurate, and highly reliable data processing. The computer software can detect inaccuracies in the instrumentation, such as a drift in flowmeter calibration. It can also be used for a variety of calculations.

## APPENDIX II

### COMPUTER PROGRAMS LISTING

```

*****
*
*   CHROMATOGRAPHY PROGRAM   *
*
*       MAIN MENU           *
*
*   KKOASH. 5th.MAY.1989   *
*
*****
COLOR 2,4
LOCATE 5,5
PRINT(CHR$(48))
CALL PROCmenu
OPEN "RUNPROG.BAT" FOR OUTPUT AS #1
STR1$ = "TBAM.BAT"
STR2$ = "TPAM.BAT"
STR3$ = "MENU.BAT"
Char$=INPUT$(1)
Char$=ucase$(Char$)
SELECT CASE Char$
  CASE "A"
    PRINT #1, STR1$
  CASE "B"
    PRINT #1, STR2$
  CASE ELSE
    COLOR 1,1
    CLS
    PRINT #1, STR3$
END SELECT
CLOSE
END

SUB PROCmenu
  LOCAL count%,i%,i2,a$
  count%=0
  CLS
  COLOR 2,4
  LOCATE 2,2 : PRINT(CHR$(201))
  FOR I=3 TO 79 : LOCATE 2,I : PRINT(CHR$(205)) : NEXT I
  LOCATE 2,79 : PRINT(CHR$(187))
  FOR I=3 TO 23 : LOCATE I,79 : PRINT(CHR$(186)) : NEXT I
  LOCATE 23,3 : PRINT(CHR$(205))
  FOR I=78 TO 2 STEP -1 : LOCATE 23,I : PRINT(CHR$(205)) : NEXT I
  LOCATE 23,79: PRINT(CHR$(188))
  LOCATE 23,2 : PRINT(CHR$(200))
  FOR I=3 TO 22 : LOCATE I,2 : PRINT(CHR$(186)) : NEXT I
  COLOR 15,4
  LOCATE 4,14
  PRINT TAB(22);"CHROMATOGRAPHY CONTROL MENU PROGRAM.";"display channels

  LOCATE 6,14
  PRINT TAB (30);"NETWORK MENU"
  LOCATE 10,14
  PRINT TAB(22);"A - Turbo Basic Chromatography Application Programs"
  LOCATE 12,14
  PRINT TAB(22);"B - Turbo Pascal Chromatography Application Programs"
  LOCATE 22,22
  COLOR 1,4
  PRINT" Please enter choice or Q to quit. "
END SUB

```

```

'*****
'*                                     *
'*   CHROMATOGRAPHY PROGRAM         *
'*                                     *
'*       MAIN MENU                   *
'*                                     *
'*   KKDASH. 5th.MAY.1989           *
'*                                     *
'*****

```

```

COLOR 15,1
LOCATE 5,5
PRINT(CHR$(48))
CALL PROCmenu
Char$=INPUT$(1)
Char$=ucase$(Char$)
SELECT CASE Char$
  CASE "A"
    CHAIN"CHPRO1"
  CASE "B"
    CHAIN"CHPRO2"
  CASE "C"
    CHAIN"CHPRO3"
  CASE "D"
    CHAIN"CHPRO4"
END SELECT

```

END

```

SUB PROCmenu
  LOCAL count%,i%,i2,a$
  count%=0
  CLS
  COLOR 2,1
  LOCATE 2,2 : PRINT(CHR$(201))
  FOR I=3 TO 79 : LOCATE 2,I : PRINT(CHR$(205)) : NEXT I
  LOCATE 2,79 : PRINT(CHR$(187))
  FOR I=3 TO 23 : LOCATE I,79 : PRINT(CHR$(186)) : NEXT I
  LOCATE 23,3 : PRINT(CHR$(205))
  FOR I=78 TO 2 STEP -1 : LOCATE 23,I : PRINT(CHR$(205)) : NEXT I
  LOCATE 23,79: PRINT(CHR$(188))
  LOCATE 23,2 : PRINT(CHR$(200))
  FOR I=3 TO 22 : LOCATE I,2 : PRINT(CHR$(186)) : NEXT I
  COLOR 15,1
  LOCATE 4,14
  PRINT TAB(22);"CHROMATOGRAPHY CONTROL MENU PROGRAM." 'display chan
  LOCATE 6,14
  PRINT TAB (30);"TURBO BASIC MAIN MENU"
  LOCATE 10,14
  PRINT TAB(22);"A - Initialise data reading" 'display time
  LOCATE 12,14
  PRINT TAB(22);"B - Read data" 'display scans
  LOCATE 14,14
  PRINT TAB(22);"C - Data processing"
  LOCATE 22,22
  COLOR 4,3
  PRINT" Please enter choice or Q to quit. "
END SUB

```



MAIN MENU - SETUP MODE

---

Parameters setup  
Analog input units and ranges setup  
Analog input scale factors setup  
Maximum / minimum / average setup  
Analog input alarms setup  
Clock setup  
Digital I/O setup  
Analog control setup  
Digital control setup  
Analog output setup  
Graphing setup  
Picture display setup  
Data log and reset setup  
Calibrate  
Save or load setup on disk  
Customizeable by programmer  
Normal display mode - exit setup mode  
Fast read mode - exit setup mode  
Exit program - erase setup  
a selection, press DY

---

IONS: MOVE CURSOR(,End) DELETE(D,Del) ERASE LINE(Home) EXIT(Esc) HELP(?)

MAIN MENU - DISPLAY MODE

---

- 1 Analog inputs display
  - 2 Maximum / minimum of analog inputs display
  - 3 Average of analog inputs display
  - 4 Difference of analog inputs display
  - 5 Analog alarms display
  - 6 Digital I/O display
  - 7 Graphing display
  - 8 Picture display
  - 9 No display (fast cycle)
  
  - A Audible alarm signal On/Off
  - C Go to setup mode
  - D Data log on disk or printer
  - G Graphing setup
  - O Switch digital outputs
  - R Reset maximum / minimum / average / control time lists
  - Z Customizeable by programmer
  - X Exit program - erase setup
-

G 08 1991 13:09:36 HRS

Present analog inputs

Input Channel 1	21.67829	Degrees C
Input Channel 2	21.80852	Degrees C
Input Channel 3	.1329416	Volts

Difference

Channel 1 - 2	-.13023	Degrees C
---------------	---------	-----------

Reset AUG 08 1991 13:09:24 HRS

Maximum / Minimum

Input Channel 1	21.67829	21.65464	Degrees C
Input Channel 2	21.82235	21.7851	Degrees C
Input Channel 3	.1330739	.1328286	Volts

Average / Number of samples

Input Channel 1	21.66	Degrees C	4
Input Channel 2	21.8	Degrees C	4
Input Channel 3	.1329402	Volts	4

Digital I/O

I/O 1	high
I/O 2	high
I/O 3	high
I/O 4	high
I/O 5	high
I/O 6	high
I/O 7	high
I/O 8	high
I/O 9	high
I/O 10	high
I/O 11	high
I/O 12	high

G 08 1991 13:37:30 HRS

Present analog inputs

Input Channel 1	21.80184	Degrees C
Input Channel 2	21.90434	Degrees C
Input Channel 3	.1335007	Volts

Difference

Channel 1 - 2	-.1025047	Degrees C
---------------	-----------	-----------

Reset AUG 08 1991 13:36:30 HRS

Maximum / Minimum

Input Channel 1	21.80184	21.76121	Degrees C
Input Channel 2	21.93089	21.90434	Degrees C
Input Channel 3	.133591	.1335007	Volts

Average / Number of samples

Input Channel 1	21.79	Degrees C	4
Input Channel 2	21.92	Degrees C	4
Input Channel 3	.1335533	Volts	4

Digital I/O

I/O 1	high
I/O 2	high
I/O 3	high
I/O 4	high
I/O 5	high
I/O 6	high

# CHROMATOGRAPHY PROGRAM

## DATA LOGGING PROGRAM

K. KASH. 10th.MAY.1980

The following data for the machine code is improved over the example in the manual. This code checks for the presence of the driver before trying to use it.

TA &H50,&HE8,&H18,&H00,&H3D,&HFF,&HFF,&H74,&H00,&H68,&HFA,&HB8,&H59,&H47,&H60,&H90,&H90,&H0A,&H06,&H00,&HB8,&HFF,&HFF,&H5D,&H0A,&H06,&H00,&H56,&H06,&H00,&H00,&H3E,&H00,&H26,&HA1,&H80,&H01,&H3D,&H00,&H00,&H74,&H1A,&H8B,&HFO,&H

TA &HA1,&H82,&H01,&H3D,&H00,&H00,&H74,&H0F,&H3E,&H00,&H26,&H8A,&H04,&H30,&H75,&H06,&H07,&H5E,&HB8,&H00,&H00,&H03,&HB8,&HFF,&HFF,&H07,&H5E,&H03,0

%=8:SM%=15:DIM B(15),A%(15):REM Allow for 16 chans & I/O's

OSUB 50000: REM Initial setup

DEF SEG:REM Set resolution to 18 bits (low noise mode):

CS="a"+CHR\$(0):A%(0)=13:CALL AM(A%(0),B(0),CS)

OSUB 51000:REM Specify range on each analog channel

OSUB 52000:REM Set up logging

REM Now read data

DEF SEG:CS="cm"+CHR\$(0):CALL AM1(A%(0),B(0),CS):REM Read analog into B()

DEF SEG:CS="I"+CHR\$(0):CALL AM1(A%(0),B(0),CS):REM Read digital into A%()

OSUB 1000:REM Display readings

REM Log data

DEF SEG:CS="1 "+CHR\$(0):CALL AM2(B(0),B(0),B(0),B(0),A%(0),B(0),B(0),CS)

\$=INKEY\$:IF A\$<>CHR\$(27) THEN 210

OSUB 52100:REM Turn off data logging

END

REM \*\*\* DISPLAY SUBROUTINE \*\*\*

CLS:PRINT "Press 'Esc' to stop":PRINT

PRINT "CHANNEL ANALOG INPUTS DIGITAL INPUTS"

FOR A=0 TO SN%-1:PRINT A+1,B(A)::LOCATE ,32:PRINT A%(A):NEXT A

RETURN

REM \*\*\* INITIAL SETUP SUBROUTINE \*\*\*

DEF SEG:APROG\$=SPACE\$(80):APROG1\$=SPACE\$(80):REM Strings will contain routines to CALL driver

AX=VARPTR(APROG\$):AX%=VARPTR(APROG1\$)

AM1=PEEK(AX+1)+PEEK(AX+2)\*256:AM2=PEEK(AX%+1)+PEEK(AX%+2)\*256

RESTORE 60

FOR A=0 TO 76

READ AX:POKE A-AM1,AX:POKE A-AM2,AX:REM Install routines to CALL driver

NEXT:POKE AM2+19,15:POKE AM2+26,15

CS="Fn"+CHR\$(0)

CALL AM1(A%(0),B(0),CS):PRINT:REM Get no. of chans & I/O's installed

IF A%(0) = 0 AND A%(2) = 0 THEN PRINT"Driver, ADRIVE.COM, not installed, no log card not installed.":END

IF A%(0) = 0 AND A%(2) <> 0 THEN PRINT"No analog card selected. BRD SEL is set to 0.":END

IF A%(0) <> 0 AND A%(6) = 0 THEN PRINT"CALIB.DAT file not correct or FIND, as not run.":END

IF A%(0) > A%(6) THEN PRINT"Calibration numbers are not correct.":END

IF A%(0) > 16 OR A%(1) > SM% + 1 THEN PRINT"Too many channels installed.

e DIM statement on line 70.":END

RETURN

REM \*\*\* SUBROUTINE TO SET RANGE ON EACH CHANNEL, IN OR OUT ON EACH I/O \*\*\*

FOR A=0 TO SN% - 1

AX(A)=2:REM RANGE - 0=50MV, 1=500MV, 2=10V, 3=+/-25MV, 4=+/-250MV, 5=+/-

```

) NEXT A
) C$="rc"+CHR$(0):CALL AM1(A$(0),B(0),C$):REM Set ranges & calibrate
) FOR A=0 TO SM%
)   A$(A)=0:REM 0 INPUT, 1 OUTPUT
) NEXT A
) C$="s"+CHR$(0):CALL AM1(A$(0),B(0),C$):REM Set I/O's to input or output
) RETURN
) REM *** SET UP LOGGING ***
) B(0)=2:REM Log on disk
) B(1)=0:REM Log on each call
) B(2)=0:REM Log immediately
) B(3)=1:REM Log present analog data only
) B(4)=0:REM Log all channels once
) F$="TEST.DAT":REM file name
) C$="L"+CHR$(0)+F$+CHR$(0):CALL AM1(A$(0),B(0),C$):REM Set up logging
) RETURN
) REM *** TURN OFF LOGGING ***
) B(0)=0:REM Don't log
) C$="L"+CHR$(0)+F$+CHR$(0):CALL AM1(A$(0),B(0),C$):REM Set up logging
) RETURN
) REM LCG V3.41 06-13-89

```

DACS>dir go.bat

Volume in drive A is ICS\_DSS  
Directory of A:\DACS

BAT 936 18-03-87 4:21p  
1 File(s) 22528 bytes free

DACS>go

DACS>ECHO OFF

and Program copyright STC GGY 1986,1987,1988

Version 7 March 1989

TESTING AND CALIBRATING ANALOG INPUT.

Base Address = 0300 No Clock Cards found = 1

Address	Type	Analog inputs	Reso- lution	Analog outputs	Reso- lution	Digital I/O's
1300	Analog in	8	16	0		12

Neither BASICA.COM nor GWBASIC.EXE program was found.

Insert a disk containing BASIC and type either 'BASICA' or 'GWBASIC'

to run your version. Then insert the program disk and type 'RUN "ASTART"'.  
DACS>

REM COPYRIGHT 1984, 1985, 1986, 1987 BY WALTER MACLAY

CLEAR:APROG\$=SPACE\$(80):APROG1\$=SPACE\$(80)

DIM AM\$(12),AA\$(15),AB\$(15),AC\$(30),AD\$(15),AE\$(30),AF\$(15),AG\$(120),AX\$(15),AY\$(15),AZ\$(15),

AO\$(15),AP\$(15),AQ\$(15),AR\$(15)

ON ERROR GOTO 14020:A=TIMER:ON ERROR GOTO 0

DATA &H50,&HE8,&H18,&H00,&H3D,&HFF,&HFF,&H74,&H0C,&H58,&HFA,&HB8,&H59,&H47,&H

&H60,&H90,&H90,&HCA,&H06,&H00,&HB8,&HFF,&HFF,&H5D,&HCA,&H06,&H00,&H56,&H06,&H

&H00,&H00,&H8E,&HCO,&H26,&HA1,&H80,&H01,&H3D,&H00,&H00,&H74,&H1A,&H8B,&HFO,&H

DATA &HA1,&H82,&H01,&H3D,&H00,&H00,&H74,&H0F,&H8E,&HCO,&H26,&H8A,&H04,&H3C,&H

&H75,&H06,&H07,&H5E,&HB8,&H00,&H00,&HC3,&HB8,&HFF,&HFF,&H07,&H5E,&HC3,0

GOTO 50010: REM SETUP

A\$="ASETUP.BAS":CHAIN A\$,ALL

10 BEEP:CLS:PRINT "Insufficient memory.":END

20 RESUME 14030

30 BEEP:CLS:PRINT "Wrong version of BASIC or GWBASIC.":END

50 PRINT " Turn the power off and back on." : END

60 CLS:PRINT "Driver not installed. Can't continue.":RETURN

70 CLS:BEEP:PRINT "No card found. Can't proceed.":PRINT "See manual or to t

again.":GOTO 14150

80 CLS:BEEP:PRINT "FIND not run. Can't proceed.":PRINT "Exit BASIC and run

or type 'GO'.":RETURN

90 REM LOAD PICTURE SEUTP

10 IF PP=1 THEN PP=0:ERASE PT%,PV%,PH%,PS%,PU%,PG%

20 INPUT £1,PP:INPUT £1,PM:INPUT £1,PC:INPUT £1,PG:INPUT £1,PB\$

30 IF PP=0 OR SN%<0 THEN 32970

40 DIM PT%(SN%),PV%(SN%),PH%(SN%),PS%(SM%),PU%(SM%),PG%(SM%)

50 FOR A=0 TO SN%:INPUT £1,PT%(A):INPUT £1,PV%(A):INPUT £1,PH%(A):NEXT A

60 FOR A=0 TO AY%:INPUT £1,PS%(A):INPUT £1,PU%(A):INPUT £1,PG%(A):NEXT A

70 RETURN

80 REM \*\*\*LOAD AUTO SETUP FILE\*\*\*

90 INPUT £1,A%,AX%,AY%,AV,AW,AX,AY,AZ%,AZ:IF A%+1>AJ% THEN AW%=1:RETURN:REM

MANY CHANS IN SETUP

85 IF AY%>SM% THEN AW%=1:RETURN:REM TOO MANY I/O'S IN SETUP

90 SN%=A%:GOSUB 52210:GOSUB 50510:INPUT £1,SA,SA\$,SD,SD\$,SE\$,SE\$,SF\$,SH\$,SL\$

SQ\$,SR\$:IF SN%<0 THEN SQ\$="":GOTO 33772

0 IF AV = 1 AND SB = 0 THEN SB = AV: DIM AA(SN%)

0 IF AX = 1 AND SH = 0 THEN SH = AX: DIM SH(SN%),SL(SN%),AH%(SN%)

0 IF AX = 0 AND SH = 1 THEN FOR A = 0 TO SN%:SH(A) = 9.999999E+37:SL(A) =

9999999E+37: NEXT A

```

50 IF AY = 1 AND SM = 0 THEN SM = AY: DIM AH(SN%),AL(SN%)
50 IF AZ = 1 AND SS = 0 THEN SS = AZ: DIM SO(SN%),SS(SN%)
70 IF AZ = 0 AND SS = 1 THEN FOR A = 0 TO SN%:SO(A) = 0:SS(A) = 1: NEXT A
72 IF AW = 1 AND SC = 0 THEN SC = AW: DIM SC(SM%),SC%(SM%),SD(SM%),SS%(SM%)
74 IF SC = 1 THEN FOR A = 0 TO SM%:SC(A) = 0:SD(A) = 0:SC%(A) = - 1:SS%(A)
: NEXT A
80 IF AZ% = 1 AND SP% = 0 THEN SP% = AZ%: DIM SH%(SM%),SM%(SM%),SP%(SM%)
90 IF SP% = 1 THEN FOR A = 0 TO SM%:SH%(A) = 0:SM%(A) = 0:SP%(A) = - 1: NE
96 INPUT £1,SJ%,SI%,SJ%,SK%,SL%,SK%,SL
97 IF SI>0 THEN DIM SJ(SI,SJ%-SI%),SJ%(SI,SJ%-SI%)
98 IF SK>0 THEN DIM SK(SK,SL%-SK%),SI(SK,SL%-SK%)
00 IF SN%<0 THEN 33834
05 FOR A = 0 TO SN%
10 INPUT £1,SA$(A),SR%(A),SU$(A): IF AX = 1 THEN INPUT £1,SH(A),SL(A)
20 IF AZ = 1 THEN INPUT £1,SO(A),SS(A)
30 NEXT A
34 IF AY%<0 THEN 33870
36 FOR A = 0 TO AY%
40 INPUT £1,SA%(A),SD$(A),SV%(A): IF AW = 1 THEN INPUT £1,SC(A),SC%(A),SD(
S%(A)
50 IF AZ% = 1 THEN INPUT £1,SH%(A),SM%(A),SP%(A)
54 IF SI>0 AND A>=SI% AND A<=SJ% THEN FOR AX=0 TO SI-1:INPUT £1,SJ(AX,A-SI%
k(AX,A-SI%):NEXT AX
56 IF SK>0 AND A>=SK% AND A<=SL% THEN FOR AX=0 TO SK-1:INPUT £1,SK(AX,A-SK%
(AX,A-SK%):NEXT AX
60 NEXT A: FOR A = 0 TO AY%: IF SV%(A) = 1 THEN AV%(A) = 1:NEXT A
70 INPUT £1,GA%,GS%,HZ%,GH,GD%,G1%,GL%,GC%,GB%,GB$,GH$,G1$,GL$,GZ$,GE%
75 IF HZ%=1 AND GZ%=1 THEN ERASE GZ: DIM GZ(GL%-G1%+1,4):GOTO 33882
77 IF HZ%=1 AND GZ%=0 THEN DIM GZ(GL%-G1%+1,4):GZ%=HZ%:GOTO 33882
79 IF HZ%=0 AND GZ%=1 THEN ERASE GZ:GZ%=HZ%
81 GOTO 33883
82 FOR HH%=0 TO (GL%-G1%):FOR HX%=0 TO 3:INPUT £1,GZ(HH%,HX%):NEXT HX%:NEXT
83 INPUT £1,ST:GOSUB 32910:INPUT £1,SA%,SB%,SF$
85 INPUT £1,A%,SV,SW,SV%,SW%:IF A%<0 THEN 33896
86 IF A%>SX% THEN AW%=1:RETURN:REM TOO MANY ANA OUTPUTS IN SETUP
87 IF SV>0 THEN DIM SV(SV,SW%-SV%),SW(SV,SW%-SV%)
90 FOR A=0 TO A%:INPUT £1,SQ%(A),SW$(A)
92 IF SV>0 AND A>=SV% AND A<=SW% THEN FOR AX=0 TO SV-1:INPUT £1,SV(AX,A-SV%
(AX,A-SV%):NEXT AX
94 NEXT A
96 RETURN
00 REM FILE EXISTS? A=0 YES,1 NO,2 DISK ERR,A$=FILE.
00 A = 0: ON ERROR GOTO 33950
00 OPEN "I",£1,A$:CLOSE £1:ON ERROR GOTO 0: RETURN
00 A=0:AX = ERR:AW%=1:IF AX <> 53 THEN A = 2:CLOSE £1:RESUME 33970:REM ERROR
00 A = 1:RESUME 33970:REM NO FILE FOUND
00 ON ERROR GOTO 0:RETURN
00 REM ***SETUP***
00 GOSUB 51010
00 DEF SEG=0:A=(&H180):AX=PEEK(A)+PEEK(A+1)*256:AY=PEEK(A+2)+PEEK(A+3)*256:I
00 AND AY=0 THEN GOSUB 14160:BEEP:END:REM IS DRIVER INSTALLED?
00 DEF SEG:A%=VARPTR(APROG$):AX%=VARPTR(APROG1$):AM=PEEK(A%+1)+PEEK(A%+2)*25
=PEEK(AX%+1)+PEEK(AX%+2)*256
5 RESTORE 20:FOR A=0 TO 76:READ A%:POKE A+AM,A%:POKE A+AM1,A%:NEXT:POKE AM1
6:POKE AM1+26,16:A0$=CHR$(0):REM INSTALL ROUTINE TO CALL DRIVER
0 A$="Fn"+A0$:CALL AM(A%(0),AX%(0),A$):IF A%(0)=0 AND A%(1)=0 THEN CLS:GOSUB
70:REM Get no. of chans & I/O's installed.
5 IF A%(2)=0 THEN GOSUB 14180:END
0 AJ%=A%(0):SN%=AJ%-1: SX%=A%(1)-1: SM%=A%(2)-1: AN=A%(7):REM AJ%=No. of chans
alled, AN=addr of 1st card w. clock, SN%=No. of AI chans in use -1, SM%=No.
/O's -1, SX%=No. of AO chans in use -1
0 AW%=0:GOSUB 55010:REM Load cal nos.
0 SF%=33:SR%=18:AI%=A%(3):REM Resolution & filter delay, Max resol is determ
by the 1st card.

```

```

80 SA%=7:SB%=0:REM Default Foregrnd & Backgrnd colors
90 DEF SEG=0:AP=(PEEK(&H410) AND &H30)/16:DEF SEG:REM AP=TYPE OF DISPLAY ADA
100 AG$="A":GOSUB 52010:IF AW%=0 THEN GOTO 50300
110 GOSUB 52210:REM DIM ARRAYS
200 REM SETUP ANALOG INPUT DEFAULTS
210 IF SN%<0 THEN 50250
215 FOR A% = 0 TO SN%
220 AY$ = "          Input Channel": GOSUB 53010:SA$(A%) = AY$:SU$(A%) = "Volts
%(A%)=0
230 NEXT A%
240 REM SETUP ANALOG OUTPUT DEFAULTS
250 SF$="fff,fff.fffff":IF SX%<0 THEN 50290
255 FOR A%=0 TO SX%
260 AY$ = "          Output Channel": GOSUB 53010:SW$(A%) = AY$
270 NEXT A%
290 GOSUB 50510:REM SET DIGITAL DEFAULTS
300 GOSUB 53510:REM SETUP DRIVER
310 TY$ = "1985":AM$(1) = "JAN ":AM$(2) = "FEB ":AM$(3) = "MAR ":AM$(4) = "AP
AM$(5) = "MAY ":AM$(6) = "JUNE":AM$(7) = "JULY":AM$(8) = "AUG ":AM$(9) = "SE
AM$(10) = "OCT ":AM$(11) = "NOV ":AM$(12) = "DEC "
20 COLOR SA%,SB%
50 A$="n"+AO$:CALL AM(A%(0),AX%(0),A%):IF A%(6)<SN%+1 OR A%(4)<SX%+1 THEN AW
60 IF SN%<0 THEN 50400
70 FOR A=0 TO SN%:IF SR%(A)>19 AND A%(5)<SN%+1 THEN AW%=1:REM Chk for T/C ca
s. if needed
80 NEXT A
90 GOSUB 56010:REM Initialize graphing
40 IF AW%=0 THEN AS$="AUTOBOOT.SET":GOTO 53910:REM Go to display mode, AUTOB
SET & CALIB.DAT OK
50 GOTO 200
00 REM SETUP DIGITAL DEFAULTS
10 SH$ = "high":SL$ = "low": FOR A% = 0 TO SM%
20 AY$ = "          I/O": GOSUB 53010:SD$(A%) = AY$
30 NEXT A%
40 RETURN
00 REM ***PRINT TITLE
10 KEY OFF:CLS
20 LOCATE 12,26:PRINT"    ANALOG CONNECTION PC"
30 LOCATE 13,25:PRINT "INDUSTRIAL COMPUTER SOURCE "
00 LOCATE 1,1 : RETURN
00 REM ***LOAD 'AUTOBOOT' FILE
10 A$="AUTOBOOT.SET":GOSUB 33930:IF A <> 0 THEN AW%=1:RETURN
20 ON ERROR GOTO 33950
30 OPEN "I",#1,A$:INPUT #1,AZ$
40 IF AZ$ <> "ANALOG SETUP" THEN AW%=1:GOTO 52080
50 GOSUB 33680
60 CLOSE #1:ON ERROR GOTO 0
00 RETURN
00 REM DIM VARIABLE ARRAYS
10 IF SN%>-1 THEN DIM AV(SN%),SR%(SN%),SU$(SN%),SA$(SN%)
20 IF SM%>-1 THEN DIM AV%(SM%),SV%(SM%),SD$(SM%),SA%(SM%),AL%(SM%)
30 IF SX%>-1 THEN DIM SQ%(SX%),AW(SX%),SW$(SX%)
40 RETURN
00 REM ***DEFAULT NAME
10 AY$ = AY$+STR$ (A% + 1):RETURN
00 REM ***CALL's to set up
10 IF SN%>-1 THEN A$="N"+AO$:A%(0)=SN%+1:CALL AM(A%(0),A(0),A%):REM Chans.
20 IF SX%>-1 THEN A$="b"+CHR$(0):CALL AM(SQ%(0),A(0),A%):REM Ana output range
30 RETURN
00 REM ***EXIT TO DISPLAY
10 A$="AENDSET.BAS":CHAIN A$,,ALL
00 REM ***LOAD CAL FILE

```



```

010 DEF SEG:A$="B"+AO$:CALL AM(A%(0),A%(0),A$):AZ%=0:FOR A=0 TO 14:IF A%(2+A
>0 THEN AZ%=AZ%+1:REM AZ%=no. cards with analog inputs
015 NEXT A:IF SN%<0 THEN RETURN
020 A$="CALIB.DAT":GOSUB 33930:IF A<>0 THEN AW%=1:RETURN
030 ON ERROR GOTO 33950
040 OPEN "I",£1,A$:INPUT £1,AZ$
050 IF AZ$ <> "CALIBRATION" THEN AW%=1:GOTO 55070
060 LINE INPUT £1,AC$:INPUT £1,AY$:A=0
070 IF A%(2+A*8)=0 THEN 55090
080 INPUT £1,AD%(A):INPUT £1,AA%(A):INPUT £1,AB%(A):INPUT £1,AF%(A):INPUT £
%(A*2):INPUT £1,AC%(A*2):INPUT £1,AE%(A*2+1):INPUT £1,AC%(A*2+1):AZ%=AZ%-1
090 A=A+1:IF AZ%>0 THEN 55070
100 CLOSE £1:ON ERROR GOTO 0
110 RETURN
000 '** INITIALIZE GRAPHING
010 IF AW%=0 THEN RETURN ELSE GC%=-1:RETURN
999 REM ASTART V3.6 03-13-88

```

```

EM COPYRIGHT 1984, 1985, 1986, 1987 BY WALTER MACLAY
IF AM=0 THEN BEEP:PRINT "RUN ASTART to start the program":END
GOSUB 14630:REM SET UP LOGGING
PRINT :IF H10<>1 AND AA$="" THEN GOSUB 50010:REM SETUP
GOTO 40005: REM MAIN MENU
9 REM ***I/O SUBROUTINES***
0 ***ARRAY LOADING SECTION FOR GRAPHING
0 IF AKX=0 THEN GOSUB 46610:HHX=0 'GET VALUE FOR LAST PLOT TIME
5 GOSUB 3310 'GET ELAPSED TIME
0 IF HUX=0 THEN RETURN
5 GOSUB 3045 'LOAD ARRAYS
0 GOSUB 3350 'RESET LAST PLOT TIME
0 RETURN
5 HKX=HKX+1:IF HKX>HMX-5 THEN HKX=0:HJX=1
0 FOR HHX=0 TO GLX-G1X:H9=GZ(HHX,1):H0=GZ(HHX,2)
0 A=184-((AV(HHX+G1X-1))-(H0))/((H9)-(H0))*184
0 IF A>185 THEN A=186 ELSE IF A<0 THEN A=-1
0 HDX (HKX,HHX)=A
0 IF GSX=0 OR SC=0 THEN 3150
0 FOR HLX=0 TO SMX:IF SCX(HLX)=(HHX+G1X-1) THEN 3100
0 NEXT HLX:GOTO 3150
0 A=184-((SC(HLX))-(H0))/((H9)-(H0))*184
0 IF A>185 THEN A=186 ELSE IF A<0 THEN A=-1
0 HSX (HKX,HHX)=A
0 NEXT HHX
0 RETURN
REM ELAPSED TIME ROUTINE
HV=TIMER:IF HSFX=1 THEN 3315 ELSE 3325
IF HV<HST THEN HUX=1:HHX=1:HSFX=0:HZZX=1:RETURN
IF HV-HZ>=GH THEN HUX=1:HHX=1:HSFX=0:RETURN
GOTO 3335
IF HV<HZ THEN HZ=HZ-86400!
IF HV-HZ>=GH THEN HUX=1:HHX=1:GOTO 3340
HUX=0
RETURN
REM RESET LAST PLOT TIME
HV=TIMER:IF HZZX=1 THEN HZZX=0:HZ=0:RETURN
IF HV<HZ OR HZ=86400! THEN HZ=HZ-86400!
HZ=HZ+GH:IF HZ+GH<HV THEN 3360
RETURN
***MAIN CONTROL FOR GRAPHING ROUTINES
IF AP<>1 AND AP<>2 THEN CLS:PRINT "Color/Graphics adaptor not installed. Can't display graphing":GOSUB 13310:AA$=""
RETURN
IF SNX<0 THEN CLS:PRINT "No analog inputs installed. Can't display graphing":GOSUB 13310:AA$="" :RETURN
IF GZX=1 THEN 3430
GOSUB 46510:AA$="" :GOTO 3499
GOSUB 3510 '* to draw the screen
IF HHX = 0 OR AKX=0 THEN AA$="":GOTO 3470
GOSUB 46010:IF AA$<> "" THEN 3490 ELSE 3470 ' plot all filled arrays
IF HUX<>0 THEN GOSUB 45010 ' plot latest values
GOSUB 10010 ' I/O cycle
IF AA$ = "" THEN 3460
WIDTH 80:SCREEN 0,1:COLOR SAX,SBX
RETURN
*** DRAW X Y AXIS
IF GDY = 2 THEN 3530
SCREEN 1,0:COLOR GBX,GCX:GOSUB 3680:GOTO 3540
SCREEN 2,0:GOSUB 3840
PSET (3,0):LINE -(3,185)
PSET (4,185):LINE -(4,0)
FOR HHX=6 TO 185 STEP 20
PSET (4,HHX):LINE -(0,HHX)
NEXT HHX
PSET (3,HHX):LINE -(HHX,HHX)
FOR HOX=HGX TO HMX STEP HGX
PSET (HOX,HHX):LINE -(HOX,HHX+2):LINE -(HOX+1,HHX+2):LINE -(HOX+1,HHX)

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NEXT H0X
LOCATE 1,1,0:VIEW SCREEN (5,0)-(HMX,184)
RETURN
'* PRINT UP TO 10 CHANNEL NUMBERS WITH COLOR BARS
HMX=1:HXX=0:HYX=G1X
IF GZ(HXX,0)=0 THEN 3700 ELSE 3710
HXX=HXX+1:HYX=HYX+1:IF HYX > GLX THEN 3810 ELSE 3690
HG$=MID$(STR$(HYX),2,3):IF LEN(HG$)<=2 THEN HG$=" "+HG$
LOCATE 25,HMX,0:PRINT HG$:LINE (0,199)-(23,199),GZ(HXX,3)
FOR HMX=31 TO 287 STEP 32
HMX=HMX+4:HXX=HXX+1:HYX=HYX+1:IF HYX > GLX+1 THEN 3810
IF GZ(HXX,0)=0 THEN 3760 ELSE 3770
HXX=HXX+1:HYX=HYX+1:IF HYX > GLX THEN 3810 ELSE 3750
HG$=MID$(STR$(HYX),2,3):IF LEN(HG$)<=2 THEN HG$=" "+HG$
LOCATE 25,HMX,0:PRINT HG$:
LINE (HMX,199)-(HMX+24,199),GZ(HXX,3)
NEXT HMX
RETURN
'* PRINT UP TO 20 CHANNEL NUMBERS
HMX=1:HXX=0:HYX=G1X
IF GZ(HXX,0)=0 THEN 3860 ELSE 3870
HXX=HXX+1:HYX=HYX+1:IF HYX > GLX THEN 3960 ELSE 3850
HG$=MID$(STR$(HYX),2,3):IF LEN(HG$)<=2 THEN HG$=" "+HG$
LOCATE 25,HMX,0:PRINT HG$
FOR HMX=1 TO (GLX-G1X+1)
HMX=HMX+4:HXX=HXX+1:HYX=HYX+1:IF HYX > GLX+1 THEN 3960
IF GZ(HXX,0)=0 THEN 3920 ELSE 3930
HXX=HXX+1:HYX=HYX+1:IF HYX > GLX THEN 3960 ELSE 3910
HG$=MID$(STR$(HYX),2,3):IF LEN(HG$)<=2 THEN HG$=" "+HG$
LOCATE 25,HMX,0:PRINT HG$:
NEXT HMX
RETURN
REM ***READ ANALOG
DEF SEG:AS$="cm"+A0$ CALL AM(AVX(0),AV(0),AS$):REM READ ANALOG INTO AV()
FOR AX=0 TO SNX:IF SS=1 THEN AV(AX)=AV(AX)*SS(AX)-SO(AX)
IF SRX(AX)<20 THEN 5200
AS$=RIGHT$(SU$(AX),1):IF AS$="F" THEN AW=AV(AX)*9/5+32:GOSUB 7210:AV(AX)=AW
IF AS$="K" THEN AV(AX)=AV(AX)+273.16
NEXT AX:RETURN
REM ROUND OFF
AW=INT(AW*100+.5)/100:RETURN
REM ***MAX/MIN/AVE/ALARM
IF SB = 0 AND SM = 0 AND SH = 0 THEN RETURN
AC = 0: FOR A = 0 TO SNX: IF SM = 0 THEN 7350
IF AV(A) > AH(A) THEN AH(A) = AV(A)
IF AV(A) < AL(A) THEN AL(A) = AV(A)
IF SB = 1 THEN AW = (AA(A) * AS + AV(A))/(AS+1):IF SRX(A) > 19 THEN GOSUB 7210
IF SB = 1 THEN AA(A) = AW
IF SH = 0 THEN 7410: REM ALARMS
IF AV(A) > = SH(A) THEN AHX(A) = 2:AC = 1: GOTO 7410
IF AV(A) < = SL(A) THEN AHX(A) = 1:AC = 1: GOTO 7410
AHX(A) = 0
NEXT:IF SB=1 THEN AS=AS+1
RETURN
REM ***READ DIGITAL
DEF SEG:AS$="I"+A0$ CALL AM(AVX(0),A(0),AS$)
RETURN
REM CHK DIGITAL ALARMS
AD = 0:FOR A = 0 TO SMX:IF SA$(A) = 0 THEN 8080
IF AVX(A) = SA$(A) - 1 THEN ALX(A) = 1:AD = 1:GOTO 8080
ALX(A) = 0
NEXT A: RETURN
REM ***CNTRL
IF SC = 0 AND SPX = 0 THEN RETURN
FOR AY = 0 TO SMX:AX = AVX(AY):AZ=INT(AY/8)*8
REM CNTRL BY ANALOG

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IF SC=0 OR SNX<0 THEN 9110
IF SCX(AY) = - 1 THEN 9110
IF AV(SCX(AY)) > SC(AY) + SD(AY) THEN AX = SSX(AY): REM ACTIVE
IF AV(SCX(AY)) < SC(AY) - SD(AY) THEN AX = 0: IF SSX(AY) = 0 THEN AX = 1: REM INACTIVE
REM CNTRL BY DIGITAL
IF SPX = 0 THEN 9190
IF SPX(AY) = - 1 THEN 9190
AX = 0: IF SHX(AY) = 0 THEN AX = 1: REM INACTIVE
AZX = AX: GOSUB 9510: FOR AX = 0 TO 7
  IF AXX(AX) = 0 THEN 9180
  IF AVX(AX + AZ) <> AX(AX) THEN AX = 7: AX = AZX: GOTO 9190: REM INACTIV
  AX = SHX(AY): REM ACTIVE
NEXT AX
AVX(AY)=AX:NEXT AY:GOSUB 9300
RETURN
DEF SEG:AS="0"+A0$ CALL AM(AVX(0),A(0),A$):REM Set I/O's
RETURN
REM SEP CNTRL BITS
A = (SPX(AY) + 1) / 256:AW = (SMX(AY) + 1) / 256
FOR AX = 7 TO 0 STEP - 1:AX(AX) = 0:A = A * 2: IF A > 1 THEN A = A - 1:AX(AX) = 1
  AXX(AX) = 0:AW = AW * 2: IF AW > 1 THEN AW = AW - 1:AXX(AX) = 1
NEXT AX: RETURN
REM ***I/O CYCLE***
AA$=INKEY$:IF AA$<>" " THEN 10110
IF SNX>-1 THEN GOSUB 5110:GOSUB 7310
AA$=INKEY$:IF AA$<>" " THEN 10110
GOSUB 8010: GOSUB 9010: GOSUB 8050
IF GZX=1 AND SNX>-1 THEN GOSUB 3010
GOSUB 11010: IF (AC = 1 OR AD = 1) AND ACX = 0 THEN BEEP
AA$=INKEY$:IF AA$<>" " THEN 10110
AKX=1:GOSUB 10210
RETURN
AS=MID$(AA$,2,1):IF AS="I" OR AS="Q" THEN AA$=AS:GOSUB 37010:AA$=""
RETURN
REM ***DATALOG/RESET/TIME LIST***
GOSUB 10510:GOSUB 11410
REM LOG DATA
IF SD = 0 OR AKX = 0 THEN 10310
AS="1 "+A0$:DEF SEG:A=1+SM*2+SB*4:ON A GOSUB 10280,10280,10285,10285,10290,10290,10295
IF MID$(AS,3,1)="2" THEN GOSUB 14750:BEEP:LOCATE 25,1:COLOR SBX+16,SAX:PRINT AES;:COLOR SAX,SBX:LOCATE 1
IF (SD=1 OR SD=3) AND MID$(AS,3,1)="1" THEN 10850
GOTO 10310
CALL AM1(AV(0),AV(0),AV(0),AV(0),AVX(0),AV(0),AV(0),A$):RETURN
CALL AM1(AV(0),AH(0),AL(0),AV(0),AVX(0),AV(0),AV(0),A$):RETURN
CALL AM1(AV(0),AV(0),AV(0),AA(0),AVX(0),AV(0),AV(0),A$):RETURN
CALL AM1(AV(0),AH(0),AL(0),AA(0),AVX(0),AV(0),AV(0),A$):RETURN
REM DATALOG ON ALARM
IF SA = 0 OR AKX = 0 THEN 10400
IF AC = 0 AND AD = 0 THEN ADX = 0: GOTO 10400
IF ADX > 0 THEN 10400
ADX=1:IF SA=1 OR SA=3 THEN GOSUB 10850:IF SA=1 THEN 10400:REM LOG ON PRINTER
IF SA$<>SD$ THEN AF$ = SA$:AWX = SA:GOSUB 10830:GOTO 10400:REM ALARMS IN SEPARATE LOG FILE
AS="K "+A0$:DEF SEG:A=1+SM*2+SB*4:ON A GOSUB 10280,10280,10285,10285,10290,10290,10295:REM ALARMS IN REGULAR LOG
IF MID$(AS,3,1)="2" THEN GOSUB 14750:BEEP:LOCATE 25,1:COLOR SBX+16,SAX:PRINT AES;:COLOR SAX,SBX:LOCATE 1
REM AUTO RESET
GOSUB 10510:A = 0
IF SQ$ = "" THEN RETURN
IF AQ > AW THEN RETURN
AY = AQ:AX$ = SQ$: GOSUB 10710:AQ = AY: GOSUB 39270
A=1:RETURN
REM UPDATE AQ, AZ=PRESENT HR, AW=PRESENT TIME (SEC) PAST THE HR, AH=HRS SINCE ENTERED DISPLAY MODE
IF SD = 0 AND SQ$ = "" AND SV=0 AND SI=0 AND SK=0 THEN RETURN
AZ=VAL(MID$(TI$,14,2)):AW=VAL(MID$(TI$,17,2))*60+VAL(MID$(TI$,20,2))
IF AZ=AHX THEN RETURN
AY=12:IF ST=2 THEN AY=24

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0 IF AH%>AZ THEN 10600
0 IF SQ$ <> "" THEN AQ = AQ - (AZ - AH%) * 3600
0 AH=AH+AZ-AH%:AH% = AZ: RETURN
0 IF SQ$ <> "" THEN AQ = AQ - (AZ - AH% + AY) * 3600
0 AH=AH+AZ-AH%+AY:AH% = AZ: RETURN
0 REM NEW INTERVAL. AY=INTERVAL IN SEC
0 AX = VAL (AX$):A = 1:AX$ = RIGHT$ (AX$,3): IF AX$ = "MIN" THEN A = 60
0 IF AX$ = "HRS" THEN A = 3600
0 IF AX$ = "AYS" THEN A = 86400!
0 AY = AY + AX * A: IF AY < AW THEN 10740
0 RETURN
0 REM DATALOG
0 IF AW% = 1 OR AW% = 3 THEN GOSUB 10850
0 IF AW% < 2 THEN RETURN
5 REM DATALOG ON DISK
0 GOSUB 10950:A=AV:IF AV=2 THEN RETURN:REM A=1 IF FILE NOT EXIST
5 AV=0:ON ERROR GOTO 10910:GOSUB 33430:IF AV=1 THEN AES="Tried to write to non-datalog file"
7 RETURN
0 REM DATALOG ON PRNTR
0 AV=0:ON ERROR GOTO 10910:OPEN "O",#1,"LPT1":AS = "P": GOSUB 42010: CLOSE #1:ON ERROR GOTO 0:RETURN
0 GOSUB 33930: IF A #2 THEN GOSUB 31010:RETURN
0 ON ERROR GOTO 10910:GOSUB 33430:RETURN
0 REM ERR ROUTINE FOR AUTOMATIC DATALOG. ERR MSSG = AES
0 GOSUB 31010:RESUME 10930
0 AV=2:ON ERROR GOTO 0:BEEP:LOCATE 25,1:COLOR SBX+16,SAX:PRINT AES;:COLOR SAX,SBX:LOCATE 1
0 RETURN
0 REM FILE EXISTS? (FOR AUTO DATALOG) AV=0,YES, 1,NO, 2,ERR
0 AV=0:AX=0:ON ERROR GOTO 10970:OPEN "I",#1,AF$:CLOSE #1:ON ERROR GOTO 0:RETURN
0 GOSUB 31010:AX=ERR:RESUME 10980
0 AV=2:IF AX=53 THEN AV=1:AES=""
0 RETURN
0 REM ***READ TIME***
0 GOSUB 11310
0 A = VAL ( LEFT$ (AY$,2)): IF A > 12 THEN A = 12
0 TIS = AMS(A) + " " + MID$ (AY$,4,2) + " " + TY$ + " "
0 AX$ = "HRS":AX = VAL ( MID$ (AY$,7,2)): IF ST = 2 THEN GOTO 11080: REM 24 HR CLOCK
0 AX$ = "AM ": IF AX > 11 THEN AX$ = "PM "
0 IF AX > 12 THEN AX = AX - 12
0 IF AX = 0 THEN AX = 12
0 GOSUB 11210
0 TIS = TIS + AS + ":" + MID$ (AY$,10,2) + ":" + MID$ (AY$,13,2) + " " + AX$
0 RETURN
0 REM CONVERT AX TO AS W. LEAD 0
0 AS = STR$ (AX):AS=RIGHT$(AS,LEN(AS)-1)
0 IF LEN (AS) < 2 THEN AS = "0" + AS
0 RETURN
0 REM READ SYSTEM CLOCK
0 AS=DATE$:AY$=LEFT$(AS,2)+"/"+MID$(AS,4,2):TY$=RIGHT$(AS,4)
0 AY$=AY$+" "+TIME$+"0.000"
0 RETURN
0 REM Time lists
0 IF SV=0 AND SI=0 AND SK=0 THEN RETURN
0 AY=AH*3600+AW-AJ:REM AY=total elapsed time
0 IF SV=0 THEN 11600
0 REM Analog output time list
0 FOR A=SV% TO SW%:IF AW%(A-SV%)=SV OR SV(0,A-SV%)<0 THEN 11560
0 IF SV(AW%(A-SV%)+1,A-SV%) > AY-AX(A-SV%) THEN 11550
0 IF AW%(A-SV%)<SV THEN AW%(A-SV%) = AW%(A-SV%)+1:IF AW%(A-SV%)=SV AND SW=0 THEN GOSUB 11940:AX(A-SV%)=AX(A-SV%)+
-SV%:AW%(A-SV%)=0:REM Next time pt and Start new cycle
0 IF AW%(A-SV%)<SV THEN 11520
0 GOTO 11560:REM 1 cycle done, don't repeat
0 AW(A)=((AY-AX(A-SV%))-(SV(AW%(A-SV%),A-SV%)))/((SV(AW%(A-SV%)+1,A-SV%))-(SV(AW%(A-SV%),A-SV%)))*(SW(AW%(A-SV%)+
-SW(AW%(A-SV%),A-SV%))+SW(AW%(A-SV%),A-SV%):REM New set point
0 AX(A)=AW(A)*40.95
0 NEXT A
0 AX%(0)=1:AX%(1)=0:AX%(2)=SX%+1:AX%(3)=1:AX%(4)=0

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10 DEF SEG:AS$="W"+AO$:CALL AM(AXX(0),AX(0),A$):REM Set ana outputs
20 REM Change set point
30 IF SK=0 THEN 11800
40 FOR A=SK% TO SL%:IF AK%(A-SK%)=SK OR SK(0,A-SK%)<0 THEN 11700
50 IF SK(AK%(A-SK%)+1,A-SK%)>AY-AK(A-SK%) THEN 11650
60 IF AK%(A-SK%)<SK THEN AK%(A-SK%)=AK%(A-SK%)+1:IF AK%(A-SK%)=SK AND SL=0 THEN GOSUB 11980:AK(A-SK%)=AK(A-SK%)+S
-SK%:AK%(A-SK%)=0:REM Next time pt and Start new cycle
75 IF AK%(A-SK%)<SK THEN 11620
80 GOTO 11700:REM 1 cycle done, don't repeat
90 SC(A)=((AY-AK(A-SK%))-(SK(AK%(A-SK%),A-SK%)))/((SK(AK%(A-SK%)+1,A-SK%))-(SK(AK%(A-SK%),A-SK%)))*(SI(AK%(A-SK%)
X)-SI(AK%(A-SK%),A-SK%))+SI(AK%(A-SK%),A-SK%):REM New set point
100 NEXT A
110 REM Digital output time list
120 IF SI=0 THEN 11880
130 FOR A=SI% TO SJ%:IF AJ%(A-SI%)=SI OR SJ(0,A-SI%)<0 THEN 11870
140 IF SJ(AJ%(A-SI%)+1,A-SI%)>AY-AI(A-SI%) THEN 11860
150 IF AJ%(A-SI%)<SI THEN AJ%(A-SI%)=AJ%(A-SI%)+1:IF AJ%(A-SI%)=SI THEN GOSUB 11960:IF SJ=0 THEN AI(A-SI%)=AI(A-S
AX,A-SI%):AJ%(A-SI%)=0:ELSE AV%(A)=SJ%(AX,A-SI%):REM Next time pt and Start new cycle or last pt if single cycle.
160 IF AJ%(A-SI%)<SI THEN 11820
170 GOTO 11870:REM 1 cycle done, don't repeat
180 AV%(A)=SJ%(AJ%(A-SI%),A-SI%):REM New I/O value
190 NEXT A
200 IF SK<>0 OR SI<>0 THEN GOSUB 11910
210 RETURN
220 REM Set I/O's to value of AV%()
230 GOSUB 9300:GOSUB 8050:RETURN
240 AX=AW%(A-SV%):REM Now incr start time by amt of last nonzero set pt. time
250 IF SV(AX,A-SV%)=0 THEN AX=AX-1:GOTO 11950: ELSE RETURN
260 AX=AJ%(A-SI%):REM Now incr start time by amt of last nonzero time pt.
270 IF SJ(AX,A-SI%)=0 THEN AX=AX-1:GOTO 11970: ELSE RETURN
280 AX=AK%(A-SK%):REM Now incr start time by amt of last nonzero set pt. time
290 IF SK(AX,A-SK%)=0 THEN AX=AX-1:GOTO 11990: ELSE RETURN
300 REM ***INPUT ROUTINE*** AS$=INPUT; AX=POSN OF CURSOR; ON EXIT=LEN(AS); AX=H POSN OF 1ST CHAR; A=ASC(AS),LAST CHA
T; AAS$=2ND CHAR FROM INKEY$. SET BEFORE GOSUB: AYX$=MAX LNTH OF INPUT LINE; AXN=1 NUMERIC ONLY, RETURNS=2 OF NONNU
ENTRY
310 IF AXN = 2 THEN AXN = 1: GOTO 12100
320 AS$ = ""
330 REM ENTRY PT FOR AS$<>""
340 IF AXN = 2 THEN AXN = 1: GOTO 12100
350 AX = 0:AX = POS(0): IF AYX < 1 THEN AYX = 1
360 AX$ = INKEY$: IF AX$ = "" THEN 12100
370 AAS$=MID$(AX$,2,1):A = ASC (AX$): IF AX$ = CHR$(13) OR AAS$="H" OR AAS$="I" OR AAS$="P" OR AAS$="Q" THEN AX=LEN(AS)
12140: REM CR
380 IF AX$ < > CHR$(27) THEN 12200: REM ESC
390 AX = 0
400 IF AX = 0 THEN AS$ = ""
410 LOCATE ,AX+AX:RETURN
420 IF AAS$ = "G" THEN LOCATE ,AX:AX = 0:AS$ = "": GOSUB 12910:LOCATE ,AX: GOTO 12100: REM Home
430 IF AAS$ <> "K" THEN 12250: REM <-
440 IF AX = 0 THEN 12100
450 AX=AX-1:LOCATE ,AX+AX:GOTO 12100
460 IF AX$ <> CHR$(8) THEN 12280:REM Backspace
470 IF AX=0 THEN 12100
480 AS$=LEFT$(AS$,AX-1)+MID$(AS$,AX+1,LEN(AS$)-AX):AX=AX-1:LOCATE ,AX+AX,0:GOSUB 12910:LOCATE ,AX+AX,1:PRINT MID$(AS$,AX+
$)-AX):LOCATE ,AX+AX:GOTO 12100
490 IF AAS$ <> "O" THEN 12300:REM End
500 AX=LEN(AS$):LOCATE ,AX+AX:GOTO 12100
510 IF AX + AX > 79 THEN 12100
520 IF AX = AYX THEN 12100
530 IF AAS$ <> "M" THEN 12350: REM ->
540 IF AX = LEN (AS$) THEN 12100
550 LOCATE ,AX + AX + 1:AX = AX + 1: GOTO 12100
560 IF AAS$ <> "S" THEN 12400:REM Del
570 IF AX = LEN (AS$) THEN 12100
580 AS$=LEFT$(AS$,AX)+MID$(AS$,AX+2,LEN(AS$)-AX-1):GOSUB 12910:LOCATE ,AX+AX:PRINT MID$(AS$,AX+1,LEN(AS$)-AX):LOCATE ,AX+
12100

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00 IF AAS>CHR$(2) AND AAS<CHR$(133) THEN 12100
00 IF A < 32 THEN AX$ = CHR$(A + 64): REM MAKE PRINTABLE
00 PRINT AX$;:IF AX$ < > 1 THEN 12440
00 IF AX$ <> "E" AND AX$ <> "e" AND AX$ <> "." AND AX$ <> "+" AND AX$ <> "-" AND AX$ <> " " AND (A < 48 OR A > 57)
AX$ = 2: LOCATE ,AX + AX: BEEP: RETURN:REM NOT NUMERIC
00 AX = AX + 1: IF AX > LEN (A$) THEN A$ = A$ + AX$: GOTO 12100
00 IF AX = 1 THEN A$ = AX$ + MID$(A$,AX + 1): GOTO 12100
00 A$ = LEFT$(A$,AX - 1) + AX$ + MID$(A$,AX + 1)
00 GOTO 12100
00 REM CLR EOL FOR INPUT ROUTINE
00 FOR AAC=POS(0) TO AX+AY%-1:PRINT " ";:NEXT AAC:RETURN
00 REM ***INPUT 'PRESS RET'
00 GOSUB 14010:AY% = 2:AX% = 0: GOSUB 12010: RETURN
00 REM ***INPUT 'WHICH CHAN'
00 GOSUB 14020:ARX=8
00 AY%=3:AX%=0:GOSUB 12010: IF AAS = "I" OR AAS="Q" THEN GOSUB 37010:A$ = "M": RETURN
00 IF A = 27 THEN : RETURN
00 AW% = VAL (A$) - 1: IF AW% < 0 OR AW% > AZ% OR A$ = "M" THEN BEEP: LOCATE ,AX: GOTO 13120
00 RETURN
00 REM ***INPUT ERR
00 BEEP: LOCATE ,1: RETURN
00 REM ***PRESS .. TO CONT
00 BEEP: PRINT : PRINT "Press space bar to continue";
00 A$ = INKEY$: IF A$ < > " " AND A$ < > CHR$(27) THEN 13320
00 PRINT : RETURN
00 REM ***CONT OR RETURN
00 BEEP
00 A$ = INKEY$: IF A$ = CHR$(13) OR A$ = CHR$(27) OR A$ = " " THEN PRINT : RETURN
00 GOTO 13370
00 REM EOL
00 AAC=SCREEN(CSRLIN,POS(0),1):AAA=AAC MOD 16:AAB=((AAC-AAA)/16) MOD 128:REM AAA=FOREGROUND CLR, AAB=BACKGRND CLR
00 COLOR SAX,SBX:FOR AAC=POS(0) TO 79:PRINT " ";:NEXT AAC:COLOR AAA,AAB:RETURN
00 REM ***INPUT INTEGERS, A$=""
00 AX% = 1: GOSUB 12010: IF AX% = 2 THEN 13510
00 IF VAL (A$) < - 32767 OR VAL (A$) > 32767 THEN BEEP: GOTO 13510
00 COLOR SAX,SBX : RETURN
00 REM ***INPUT FILE NAME
00 LOCATE AZ,AY: COLOR SBX,SAX: GOSUB 14080: PRINT A$;: LOCATE ,AY:AX% = 0: GOSUB 12030:LOCATE, AY:GOSUB 13410:LOCA
00 COLOR SAX,SBX:IF A = 27 OR A$="" THEN RETURN
00 IF AW=1 THEN RETURN:REM ALLOW ANY CHAR
00 AZ=0:FOR AX=1 TO LEN(A$):AX$=MID$(A$,AX,1):IF (AX$<"E" OR (AX$>"") AND AX$<"-") OR (AX$>":" AND AX$<"@") OR (AX$
00 AX$<"_" OR AX$>"") AND AX$<>"\" THEN A$="The character '"+AX$+"' is not allowed.":GOTO 13890
00 IF AX$=":" OR AX$="\" THEN AZ=AX:REM AZ=POSN OF LAST ':' OR '\'
00 NEXT AX
00 IF LEN(A$)-AZ>12 THEN A$="Name too long.":GOTO 13890
00 RETURN
00 BEEP:CLS:PRINT "Invalid file name. ";A$:GOSUB 13310:A$="":A=27:RETURN
00 REM ***PRINT STRINGS***
00 LOCATE 2,1:PRINT SPACES(26);:LOCATE ,1:PRINT TIS:AX=4:RETURN
00 PRINT CHR$(17);CHR$(196);CHR$(217);:RETURN
00 PRINT"No analog inputs in use":GOSUB 13310:AAS="":RETURN
00 CLS : PRINT "No data. First read data in any numbered menu.": GOTO 13310
00 PRINT "Type selection, press ";:GOSUB 13930:RETURN
00 PRINT "Switch which output number?";: RETURN
00 PRINT " - DISPLAY MODE";: RETURN
00 LOCATE ,1: FOR AX = 1 TO 79: PRINT "-";: NEXT : RETURN
00 PRINT "OPTIONS: MOVE CURSOR("";CHR$(27);","";CHR$(26);","End) DELETE("";CHR$(17);CHR$(196);","De1) ERASE LINE(Home) E
00 ";: RETURN
00 LOCATE ,9: PRINT "MOVE TO OTHER CHANNELS (PgDn,PgUp)";:RETURN
00 PRINT "HELP(?)";: RETURN
00 AY = POS(0): FOR AX = 1 TO A: PRINT " ";: NEXT : LOCATE ,AY: RETURN
00 PRINT "----- Input Name -----";: RETURN
00 PRINT "----- I/O Name -----";: RETURN
00 CLS : GOSUB 31010:PRINT A$:PRINT:AES="":GOSUB 13310:RETURN
00 LOCATE 24,1:A = 79: GOSUB 14080: RETURN
00 PRINT "Press space bar to continue. Press ";:GOSUB 13930:PRINT " to go back.": GOSUB 13360: RETURN

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0 PRINT "Press a menu key or 'Esc' for menu";:IF SNX>15 OR SMX>15 THEN PRINT ", 'PgDn' or 'PgUp' for other channels";
5 RETURN
0 LOCATE 4,27
5 PRINT "PLEASE WAIT": RETURN
0 LOCATE 2: PRINT "Reset at: ";AR$
0 LOCATE 3: RETURN
0 PRINT "Not setup": GOTO 13310
0 REM ***MENU TOP & BOTTOM-1 LINE
0 CLS
0 LOCATE 1, INT ((80 - LEN (A$) - 13) / 2 + .6): PRINT A$;: GOSUB 14030
0 PRINT : GOSUB 14040: LOCATE 23,1: GOSUB 14040:PRINT: GOSUB 14050
0 LOCATE 3: RETURN
0 REM ***MENU TOP & BOTTOM-2 LINES
0 CLS
0 LOCATE 1, INT ((80 - LEN (A$) - 13) / 2 + .6): PRINT A$;: GOSUB 14030
0 PRINT : GOSUB 14040: LOCATE 22,1: GOSUB 14040:PRINT: GOSUB 14050: PRINT : IF SNX > 15 THEN GOSUB 14060
0 LOCATE 3: RETURN
0 REM OUTPUT MENU
0 CLS: LOCATE , INT ((80 - LEN (A$) - 13) / 2 + .6): PRINT A$;: GOSUB 14030: LOCATE 24,1: GOSUB 14150:LOCATE 25,
SBX+16,SA$:PRINT AE$;:COLOR SAX,SBX:IF AE$<>" " THEN BEEP
0 LOCATE 3,1:RETURN
0 REM ***SUBMENU BOTTOM
0 CLS : LOCATE 23,1: GOSUB 14040:PRINT: GOSUB 14050: LOCATE 13,1: RETURN
0 REM ***SET UP DATA LOGGING
0 IF SD = 0 THEN RETURN
0 A$="L"+A0$+SD$+A0$:A(0)=SD:AX$=SE$:AY=0:AW=0:GOSUB 10710:A(1)=AY:A(2)=SE$:A(4)=0:A(5)=0:A(3)=16+SM*6+SB*8:IF SNX
EN A(3)=A(3)+1
5 DEF SEG:CALL AM(AVX(0),A(0),A$):REM Set up logging
0 IF MID$(A$,3,1)<>"2" THEN RETURN
0 IF RIGHT$(A$,12) = "AUTOBOOT.SET" THEN GOSUB 14750:RETURN
0 CLS:PRINT"DISK ERROR":PRINT"Press space bar to continue and ignore the error, ";:GOSUB 13930:PRINT" to try again
0B 13380:IF A$=CHR$(13) THEN 14630
0 IF MID$(A$,3,1)="2" THEN PRINT"Logging to disk disabled. Return to setup mode to reenale logging.":GOSUB 13310
0 RETURN
0 REM ***TURN OFF DATALOG***
0 IF SD = 0 THEN RETURN
0 A$="L"+A0$+SD$+A0$:A(0)=0:A(1)=0:A(2)=0:A(3)=0:A(4)=0:A(5)=0:DEF SEG:CALL AM(AVX(0),A(0),A$):IF MID$(A$,3,1)="2"
CLS:BEEP:PRINT"Disk error was detected.":GOSUB 13310:GOTO 14720
0 RETURN
0 AE$="Disk error. Logging disabled. Go to setup mode to reenale.":RETURN
0 REM PRNT DIGITAL SCREEN
0 GOSUB 14100: LOCATE ,27: PRINT "In/Out";: LOCATE ,35: PRINT "Status";: LOCATE ,46: PRINT "Alarm if":RETURN
0 LOCATE AZ,1: PRINT SD$(A);: LOCATE ,27: IF SV$(A) = 0 THEN PRINT "Input";
0 IF SV$(A) = 1 THEN PRINT "Output";
0 RETURN
0 LOCATE ,45: IF SA$(A) = 0 THEN PRINT "None"
0 IF SA$(A) = 1 THEN PRINT SL$
0 IF SA$(A) = 2 THEN PRINT SH$
0 RETURN
0 REM PRNT CHAN NOS.
0 AX$ = STR$ (A+1):IF LEN(AX$)=2 THEN AX$=AX$+" "
0 IF LEN(AX$)=3 THEN AX$=AX$+" "
0 LOCATE ,21: PRINT AX$;: LOCATE ,36: IF AL$(A) = 1 THEN COLOR SAX+16,SBX
0 PRINT AY$: COLOR SAX,SBX : RETURN
0 REM ***ANALOG INPUT***
0 ARX=1:LOCATE ,0:A$ = "ANALOG INPUTS": GOSUB 14520:IF SNX<0 THEN 13940
0 GOSUB 14090: LOCATE ,29: PRINT "Value";: LOCATE ,44: PRINT "Units"
0 AX=4:FOR A = ANX TO AMX: LOCATE AX,1: PRINT SA$(A);:LOCATE ,43:PRINT SU$(A);:AX = AX+1:NEXT A: PRINT
0 IF AKX = 0 THEN AA$="":GOTO 15100
0 GOSUB 13920
0 FOR A=ANX TO AMX:IF SH=1 THEN IF AH$(A)>0 THEN COLOR SAX+16,SBX
0 LOCATE AX,27:IF SR$(A)<20 THEN PRINT USING SF$;AV(A); ELSE PRINT USING "###,###,###.##";AV(A);
0 COLOR SAX,SBX:AX=AX+1:NEXT A
0 GOSUB 10010
0 IF (A$="I" OR A$="Q") AND AA$="" THEN A$="":GOTO 15010

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IF AA$="" THEN 15040
RETURN
REM ***MAX/MIN***
ARX=2:LOCATE ,,0:AS$="MAXIMUM/MINIMUM ANALOG INPUTS":GOSUB 14520:IF SNX<0 THEN 13940
GOSUB 14170:GOSUB 14090:LOCATE ,29:PRINT "Maximum";:LOCATE ,45:PRINT "Minimum";
LOCATE ,60:PRINT "Units"
IF SM=0 THEN GOSUB 14190:AA$="":RETURN
AX=4:FOR A=ANX TO AMX:LOCATE AX,1:PRINT SA$(A);:LOCATE ,59:PRINT SU$(A);:AX=AX+1:NEXT A
IF AKX=0 THEN AA$="":GOTO 16100
AX=4:FOR A=ANX TO AMX:IF SH=1 THEN IF AHX(A)>0 THEN COLOR SAX+16,SBX
LOCATE AX,27:IF SRX(A)<20 THEN PRINT USING SF$;AH(A);:LOCATE ,43:PRINT USING SF$;AL(A); ELSE PRINT USING "###,###,###.##";
AH(A);:LOCATE ,43:PRINT USING "###,###,###.##";AL(A);
COLOR SAX,SBX:AX=AX+1:NEXT A
GOSUB 10010
IF AKX=0 AND AA$="" THEN 16010
IF (A$="I" OR A$="Q") AND AA$="" THEN A$="":GOTO 16010
IF AA$="" THEN 16060
RETURN
REM ***AVERAGE***
ARX=3:LOCATE ,,0:AS$="AVERAGE OF ANALOG INPUTS":GOSUB 14520:IF SNX<0 THEN 13940
GOSUB 14170:GOSUB 14090:LOCATE ,29:PRINT "Average";:LOCATE ,44:PRINT "Units";:LOCATE ,66:PRINT "No. of samples";
IF SB=0 THEN GOSUB 14190:AA$="":RETURN
AX=4:FOR A=ANX TO AMX:LOCATE AX,1:PRINT SA$(A);:LOCATE ,43:PRINT SU$(A);:AX=AX+1:NEXT A
IF AKX = 0 THEN AA$="":GOTO 17100
AX=4:FOR A=ANX TO AMX:LOCATE AX,64:PRINT SPACES(15);:IF SH=1 THEN IF AHX(A)>0 THEN COLOR SAX+16,SBX
LOCATE AX,27:IF SRX(A)<20 THEN PRINT USING SF$;AA(A); ELSE PRINT USING "###,###,###.##";AA(A);
LOCATE ,65:PRINT AS;:COLOR SAX,SBX:AX=AX+1:NEXT A
GOSUB 10010
IF AKX=0 AND AA$="" THEN 17010
IF (A$="I" OR A$="Q") AND AA$="" THEN A$="":GOTO 17010
IF AA$="" THEN 17050
RETURN
REM ***DIFF***
ARX=4:LOCATE ,,0:AS$="DIFFERENCE OF ANALOG INPUTS":GOSUB 14520:IF SNX<0 THEN 13940
GOSUB 14090:LOCATE ,29:PRINT "Difference";:LOCATE ,44:PRINT "Units"
IF SNX=0 THEN PRINT "Only one channel set up":GOSUB 13310:AA$="":RETURN
AX=4:FOR A=INT((ANX+1)/2)*2 TO INT((AMX+1)/2)*2-1 STEP 2:LOCATE AX,1:PRINT "Channel ";RIGHT$(STR$(A+1),2);" -";RIGHT$(STR$(A+2),2);
AW=0:LOCATE ,43:IF SU$(A)=SU$(A+1) THEN AW=1:COLOR SAX,SBX:PRINT SU$(A);
IF AW=0 THEN PRINT "Not the same";
COLOR SAX,SBX:AX=AX+2:NEXT A
IF AKX=0 THEN AA$="":GOTO 18100
GOSUB 13920:FOR A=INT((ANX+1)/2)*2 TO INT((AMX+1)/2)*2-1 STEP 2:IF SU$(A)<>SU$(A+1) THEN 18098
IF SH=1 THEN IF AHX(A)>0 OR AHX(A+1)>0 THEN COLOR SAX+16,SBX
LOCATE AX,26:IF SRX(A)<20 THEN PRINT USING SF$;AV(A)-AV(A+1); ELSE PRINT USING "###,###,###.##";AV(A)-AV(A+1);
COLOR SAX,SBX
AX=AX+2:NEXT A
GOSUB 10010
IF (A$="I" OR A$="Q") AND AA$="" THEN A$="":GOTO 18010
IF AA$="" THEN 18080
RETURN
REM ***ALARMS***
ARX=5:LOCATE ,,0:AS$="ANALOG INPUT ALARMS":GOSUB 14520:IF SNX<0 THEN 13940
GOSUB 14090:LOCATE ,29:PRINT "High Limit";:LOCATE ,45:PRINT "Low Limit";
LOCATE ,60:PRINT "Units"
IF SH = 0 THEN GOSUB 14190:AA$="":RETURN
AX=4:FOR A=ANX TO AMX:LOCATE AX,1:PRINT SA$(A);:LOCATE ,59:PRINT SU$(A);:AX=AX+1:NEXT A
IF AKX = 0 THEN GOSUB 14160:AA$="":GOTO 19100
GOSUB 13920:FOR A=ANX TO AMX:LOCATE AX,26:PRINT SPACES(32);:IF AHX(A)=2 THEN COLOR SAX+16,SBX
LOCATE ,27:IF SH(A)<9.999999E+37 THEN PRINT SH(A);
COLOR SAX,SBX:IF AHX(A)=1 THEN COLOR SAX+16,SBX
LOCATE ,43:IF SL(A)>-9.999999E+37 THEN PRINT SL(A);
COLOR SAX,SBX:AX=AX+1:NEXT A
GOSUB 10010
IF (A$="I" OR A$="Q") AND AA$="" THEN A$="":GOTO 19010

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0 IF AA$="" THEN 19060
0 RETURN
0 REM ***DIGITAL***
0 ARX=6:LOCATE ,,0:AS="DIGITAL I/O STATUS":GOSUB 14520:GOSUB 14810
0 AZ=4:FOR A=AP% TO AOX:GOSUB 14820:GOSUB 14840:AZ=AZ+1:NEXT A
0 IF AKX=0 THEN LOCATE 4,34:GOSUB 14165:AA$="":GOTO 20100
0 AZ=4:GOSUB 13920:FOR A=AP% TO AOX:LOCATE AZ,34:PRINT SPACES$(11):IF ALX(A)=1 THEN COLOR SAX+16,SBX
0 LOCATE AZ,34:IF AVX(A)=0 THEN PRINT SL$:
0 IF AVX(A)=1 THEN PRINT SH$:
0 COLOR SAX,SBX:AZ=AZ+1:NEXT A
0 GOSUB 10010
5 IF (AS="I" OR AS="Q") AND AA$="" THEN AS="":GOTO 20010
0 IF AA$="" THEN 20040
0 RETURN
0 REM ***PRINT ERRORS***
0 AX = ERR
0 IF AX = 6 THEN AE$= "Overflow":RETURN
0 IF AX = 7 OR AX = 14 THEN AE$= "Out of memory":RETURN
0 IF AX < 23 OR (AX > 27 AND AX < 52) THEN AX=46:GOTO 31070
0 IF AX < 28 THEN AX = AX+24
0 IF AX > 76 THEN AX = 46
0 ON AX-46 GOTO 31100,31110,31120,31300,31130,31140,31150,31160,31170,31300,31180,31190,31300,31300,31200,31210,31
220,31300,31300,31230,31240,31300,31250,31260,31270,31300,31300,31280,31290:GOTO 31300
0 AE$= "Line buffer overflow":RETURN
0 AE$= "Device timeout":RETURN
0 AE$= "Device fault":RETURN
0 AE$= "Out of paper":RETURN
0 AE$= "Bad file number":RETURN
0 AE$= "File not found":RETURN
0 AE$= "Bad file mode":RETURN
0 AE$= "File already open":RETURN
0 AE$= "Device I/O error":RETURN
0 AE$= "File already exists":RETURN
0 AE$= "Disk full":RETURN
0 AE$= "Input past end":RETURN
0 AE$= "Bad file name":RETURN
0 AE$= "Too many files":RETURN
0 AE$= "Device unavailable":RETURN
0 AE$= "Disk write protected":RETURN
0 AE$= "Disk not ready":RETURN
0 AE$= "Disk media error":RETURN
0 AE$= "Path or file access error":RETURN
0 AE$= "Path not found":RETURN
0 AE$= "ERROR": RETURN
0 REM ***STORE DATA***
0 ARX=10:AD$ = "":AA$=""
0 AS = "STORE DATA":GOSUB 14210:LOCATE 3,1:IF AKX = 0 THEN 14000
0 PRINT " *All data will be stored that is setup for display:":IF SNX>-1 THEN PRINT " Present analog input"
0 " Difference of input"
0 IF SM=1 AND SNX>-1 THEN PRINT " Maximum, minimum of input"
0 IF SB=1 AND SNX>-1 THEN PRINT " Average of input"
0 PRINT " Digital I/O":PRINT " 1 Print data on printer":PRINT " 2 Store data on disk"
0 PRINT " 3 Disk directory":PRINT " 4 Enter data log title":PRINT " 5 Return to MAIN MENU":PRINT
0 GOSUB 13010
0 IF A = 27 OR AS = "5" THEN RETURN
0 AX = VAL (AS):IF AX < 1 OR AX > 4 THEN GOSUB 13210:GOTO 33120
0 PRINT :ON AX GOSUB 33610,33310,33260,33810:GOTO 33010
0 REM DIRECTORY
0 PRINT:PRINT "Directory of which drive or path? (press ";GOSUB 13930:PRINT " to accept)":AZ=22:AY=1:AS=AG$:AYX=3
0 AW=0:GOSUB 13810:IF AS="" OR A=27 THEN RETURN
0 AG$=AS:ON ERROR GOTO 33950
0 CLS:FILES AS:ON ERROR GOTO 0:GOSUB 13310:RETURN
0 REM SAVE ON DISK
0 GOSUB 33910:IF A = 27 OR AF$ = "" THEN RETURN
0 GOSUB 14710
0 PRINT:GOSUB 33410:IF AV=1 THEN GOSUB 14120:REM ERR

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0 GOSUB 14630
0 RETURN
0 REM STORE ON DISK, AV=1 IF ERR
0 GOSUB 33930: IF A = 2 THEN RETURN
0 ON ERROR GOTO 33950
0 IF A = 1 THEN OPEN "O",£1,AF$:WRITE £1,"DATALOG":GOSUB 43510:CLOSE £1: GOTO 33460
0 OPEN "I",£1,AF$:INPUT £1,AY$:CLOSE £1: IF AY$ < > "DATALOG" THEN AV = 1
0 IF AV = 1 THEN RETURN
0 OPEN "A",£1,AF$:A$ = "D": GOSUB 42010
5 CLOSE £1:ON ERROR GOTO 0
0 RETURN
0 REM PRINT
0 PRINT "Be sure printer is on.": GOSUB 14140: IF A$ = CHR$(13) OR A$ = CHR$(27) THEN RETURN
0 ON ERROR GOTO 33950:OPEN "O",£1,"LPT1":A$ = "P": GOSUB 42010: CLOSE £1:ON ERROR GOTO 0:RETURN
0 REM DATA LOG TITLE
0 PRINT : PRINT "Enter title to be printed at the top of the data.":AZ = 22:AY = 1:A$ = AD$:AY% = 79:A = 79:AW = 1
B 13810:IF A=27 THEN RETURN:REM AW=1,ALLOW ANY CHAR.
0 AD$ = A$:RETURN
0 REM NAME
0 PRINT:PRINT "What file name? (press "":GOSUB 13930:PRINT " to accept)":AZ = 22:AY = 1:A$ = AF$:AY% = 30:A = 30:
GOSUB 13810: IF A$ = "" OR A=27 THEN RETURN : REM DW=0,NO £ ALLOWED IN 1ST CHAR.
5 AF$ = A$: RETURN
0 REM FILE EXISTS? A=0,YES, 1,NO, 2,DISK ERR, AF$=FILE, AX=ERR £
0 AX=0:AV = 0:A = 0: ON ERROR GOTO 33950
0 OPEN "I",£1,AF$:CLOSE £1:ON ERROR GOTO 0: RETURN
0 A=0:AX = ERR:IF AX < > 53 THEN A = 2:AV = 1:CLOSE £1:GOSUB 14120:RESUME 33970:REM ERROR
0 A = 1: RESUME 33970: REM NO FILE FOUND
0 ON ERROR GOTO 0:RETURN
0 REM ***CUSTOM***
0 AA$="":A$ = "CUSTOMIZEABLE MENU": GOSUB 14210: GOSUB 14130: LOCATE 4,1
0 PRINT "See manual for instructions on programming this menu": GOSUB 13310: RETURN
0 REM ***SETUP MODE***
0 AA$="":A$ = "Go to setup mode": GOSUB 14210: GOSUB 14130: LOCATE 4,1
0 PRINT "You are about to enter the setup mode.": GOSUB 14140: IF A$ < > " " THEN RETURN
0 GOSUB 14710
0 IF SI>0 THEN ERASE AJ%,AI
0 IF SK>0 THEN ERASE AK%,AK
0 IF SV>0 THEN ERASE AW%,AX
0 H10%=0:IF HEX=1 THEN ERASE HDX:HEX=0
0 IF GZX=1 AND GSX=1 AND SC=1 THEN ERASE HSX:HTX=0
0 CLS : COLOR SBX,SAX : GOSUB 14160: COLOR SAX,SBX:A$="ASETUP.BAS":ON ERROR GOTO 33950:CHAIN A$,,ALL:RETURN
0 REM CONVERT INTRVL
0 A = 1:A$ = RIGHT$(A$,3): IF A$ = "MIN" THEN A = 60
0 IF A$ = "HRS" THEN A = 3600
0 IF A$ = "AYS" THEN A = 86400!
0 AX = AX / A: RETURN
0 REM ***GO TO INACTIVE DISPLAY MODE***
0 GOSUB 14710
0 AA$=A$:CLS:GOSUB 14140:IF A$<>" " THEN AA$="":A$="":RETURN
0 CLS:COLOR SBX,SAX:GOSUB 14160: COLOR SAX,SBX:A$="ADISPSET.BAS":ON ERROR GOTO 33950:CHAIN A$,,ALL:RETURN
0 REM ***EXIT***
0 AA$="":A$ = "EXIT PROGRAM": GOSUB 14210: GOSUB 14130: LOCATE 4,1
0 PRINT "You are about to exit the program. Be sure to save your setup first.": GOSUB 14140: IF A$ < > " " THEN
GOSUB 14710
0 CLS : PRINT "To restart the program with the setup cleared,": PRINT "type RUN ";CHR$(34);"ASTART";CHR$(34);" and
";GOSUB 13930:PRINT ".":ON ERROR GOTO 0:LOCATE 20:END
0 REM ***MOVE MENU*** ARX=no. of menu on screen
0 IF SMX < 16 AND SNX < 16 THEN RETURN
0 AXX=AMX:AYX=ANX:AX=SNX:IF ARX>5 THEN AXX=AOX:AYX=APX:AX=SMX
0 IF AA$="I" AND AYX=0 THEN BEEP:RETURN
0 IF AA$="Q" AND AXX=AX THEN BEEP:RETURN
0 IF AA$="I" THEN AYX=AYX-16
0 IF AA$="Q" THEN AYX=AYX+16
0 IF AYX+15>AX THEN AYX=AX-15
0 IF AYX<0 THEN AYX=0

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0 AX%=AY%+15:IF AX%>A% THEN AX%=AX%
0 IF AR%<6 THEN AM%=AX%:AN%=AY%
0 IF AR%>5 THEN AO%=AX%:AP%=AY%
0 RETURN
0 REM Reset
0 IF SN%<0 THEN RETURN
5 AR$=TIS:AK%=0:FOR A=0 TO SN%
0 IF SM=1 THEN AH(A)=AV(A):AL(A)=AV(A)
0 IF SB=1 THEN AA(A)=AV(A):AS=1
0 NEXT A:RETURN
0 REM ***MAIN MENU***
5 LOCATE ,,1:IF AA$<>"" THEN AS=AA$:GOTO 40070
6 IF SN%>-1 THEN AX=1 ELSE AX=9
7 IF RIGHT$(AS$,12)="AUTOBOOT.SET" THEN 40100
0 AS$="MAIN MENU":GOSUB 14210:LOCATE 24,73:GOSUB 14070:LOCATE 3,1
0 PRINT " 1 Analog inputs display":PRINT " 2 Maximum / minimum of analog inputs display":PRINT " 3 Average of analog inputs display":PRINT " 4 Difference of analog inputs display":IF AC = 1 THEN COLOR SAX+16,SB%
0 PRINT " 5 Analog alarms display":COLOR SAX,SB%:IF AD = 1 THEN COLOR SAX+16,SB%
0 PRINT " 6 Digital I/O display":COLOR SAX,SB%:PRINT " 7 Graphing display":PRINT " 8 Picture display":PRINT " 9 Main menu (fast cycle)"
5 PRINT:PRINT " A Audible alarm signal On/Off":PRINT " C Go to setup mode":PRINT " D Data log on disk or printer":PRINT " G Graphing setup"
0 PRINT " O Switch digital outputs":PRINT " R Reset maximum / minimum / average / control time lists":PRINT " Z Control time by programmer":PRINT " X Exit program - erase setup":PRINT
0 GOSUB 13010
0 AE$="":AZ$="123456789ACDGOORZX?acdgorzx":FOR A = 1 TO LEN(AZ$):IF MID$(AZ$,A,1) = AS THEN AX = A:A = LE
:GOTO 40100
0 NEXT A:GOSUB 13210:GOTO 40010
0 PRINT :ON AX GOSUB 15010,16010,17010,18010,19010,20010,3410,47010,48010,35610,35010,33005,44010,35610,35610,35610,36010,41010,35610,35010,33005,44010,35610,35610,34010,36010:LOCATE ,,1:AS=AA$:IF AA$="" THEN 40010
0 IF ASC(AA$) = 27 THEN 40010
0 GOTO 40070
0 REM ***HELP-MAIN DISPLAY MENU
0 AS$="MAINDISP.HLP":GOSUB 41110:IF A=1 THEN GOSUB 14120
0 RETURN
0 REM ***HELP MENU
0 CLS:ON ERROR GOTO 33950:OPEN "I",#1,AS$
0 LINE INPUT #1,AX$:PRINT AX$:IF NOT EOF(1) THEN PRINT:GOTO 41120
0 LOCATE 25,1:CLOSE #1:ON ERROR GOTO 0
0 AS$=INKEY$:IF AS$ < > " " AND AS$ < > CHR$(27) THEN 41130
0 RETURN
0 REM OUTPUT DATA
0 AV=0:IF AS$<>"P" THEN GOTO 43010
0 PRINT #1,TIS:IF AD$<>"" THEN PRINT #1,AD$:AD$=""
0 IF SN%<0 THEN 42400
0 PRINT #1," Present analog inputs"
0 FOR A = 0 TO SN%
0 PRINT #1,SA$(A);TAB(27);AV(A);TAB(42);SU$(A):IF SH = 0 THEN 42090
0 IF AH$(A) = 1 THEN PRINT #1,"***LOW ALARM***";
0 IF AH$(A) = 2 THEN PRINT #1,"***HIGH ALARM***";
0 PRINT #1,"":NEXT A
0 IF SN%=0 THEN 42200
0 PRINT #1," Difference"
0 FOR A = 0 TO INT((SN%+1)/2)*2-1 STEP 2
0 PRINT #1," Channel " + STR$(A + 1) + " - " + STR$(A + 2);
0 IF SU$(A) <> SU$(A + 1) THEN PRINT #1,"":GOTO 42170
0 PRINT #1,TAB(27);AV(A) - AV(A + 1);TAB(42);SU$(A)
0 NEXT A
0 IF SM<>0 OR SB<>0 THEN PRINT #1," Reset ";AR$
0 IF SM = 0 THEN 42300
0 PRINT #1," Maximum / Minimum"
0 FOR A = 0 TO SN%
0 PRINT #1,SA$(A);TAB(27);AH(A);TAB(42);AL(A);TAB(57);SU$(A)
0 NEXT A
0 IF SB = 0 THEN 42400
0 PRINT #1," Average / Number of samples"

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0 FOR A = 0 TO SNX
0 PRINT E1,SA$(A);:AW=AA(A):IF SRX(A)>19 THEN GOSUB 7210
0 PRINT E1,TAB(27);AW;TAB(42);SU$(A);TAB(55);AS
0 NEXT A
0 PRINT E1," Digital I/O"
0 FOR A = 0 TO SMX
0 PRINT E1,SD$(A);:IF AVX(A) = 0 THEN AV$=SL$
0 IF AVX(A) = 1 THEN AV$=SH$
0 PRINT E1,TAB(27);AV$;:IF ALX(A) = 1 THEN PRINT E1,TAB(40);"***ALARM***";
0 PRINT E1,"":NEXT A:PRINT E1,""
0 RETURN
0 REM WRITE TO DISK (A$=D)
0 AV=0:IF AD$<>" " THEN PRINT E1,AD$:AD$ = ""
0 AX$=TIME$:AY$=DATE$:PRINT E1,VAL(RIGHT$(AY$,2));",";VAL(LEFT$(AY$,2));",";VAL(MID$(AY$,4,2));",";VAL(LEFT$(AX$,2));
:VAL(MID$(AX$,4,2));",";VAL(RIGHT$(AX$,2));",";"00";:REM DATE & TIME
5 IF SNX<0 THEN 43070
0 FOR A=0 TO SNX:PRINT E1,"";AV(A);:NEXT A
0 IF SM>0 THEN FOR A=0 TO SNX:PRINT E1,"";AH(A);:NEXT A
0 IF SM>0 THEN FOR A=0 TO SNX:PRINT E1,"";AL(A);:NEXT A
0 IF SB>0 THEN FOR A=0 TO SNX:PRINT E1,"";AA(A);:NEXT A
0 FOR A=0 TO SMX:PRINT E1,"";AVX(A);:NEXT A
0 PRINT E1,"":RETURN
0 REM WRITE COLUMN HEADINGS TO DISK
0 AX$=SE$:AY=0:AW=0:GOSUB 10710
0 A$=CHR$(34):PRINT E1,A$;"log interval (sec)";A$;",";AY;",";SNX+1;A$;"channels";A$;",";SMX+1;A$;"digital I/O's";
:A=16+SM*6+SB*8:IF SNX>-1 THEN A=A+1:REM Log interval, no. chans, type of data in file: 1=pres, 2=max, 4=min, 8=av
dig
0 PRINT E1,A:PRINT E1,A$;"Year";A$;",";A$;"Month";A$;",";A$;"Day";A$;",";A$;"Hour";A$;",";A$;"Minute";A$;",";A$;"S
A$;",";A$;"1/100 sec.";A$;
5 IF SNX<0 THEN 43585
0 PRINT E1,"";A$;"Present: chan";A$;
0 IF SM>0 THEN GOSUB 43700:PRINT E1,"";A$;"Maximum: chan";A$;
0 IF SM>0 THEN GOSUB 43700:PRINT E1,"";A$;"Minimum: chan";A$;
0 IF SB>0 THEN GOSUB 43700:PRINT E1,"";A$;"Average: chan";A$;
0 GOSUB 43700
0 PRINT E1,"";A$;"I/O";A$;
0 PRINT E1,"":REM END OF LINE
0 PRINT E1,A$;A$;",";A$;A$;",";A$;A$;",";A$;A$;",";A$;A$;",";A$;A$;",";A$;A$;",";A$;A$;
0 IF SNX<0 THEN 43650
0 GOSUB 43720
0 IF SM>0 THEN GOSUB 43720:GOSUB 43720
0 IF SB>0 THEN GOSUB 43720
0 GOSUB 43730
0 PRINT E1,CHR$(2):REM Mark end of heading
0 RETURN
0 FOR A=0 TO SNX-1:PRINT E1,"";A$;A$;:NEXT A:RETURN
0 FOR A=0 TO SMX-1:PRINT E1,"";A$;A$;:NEXT A:RETURN
0 FOR A=0 TO SNX:PRINT E1,"";A+1;:NEXT A:RETURN
0 FOR A=0 TO SMX:PRINT E1,"";A+1;:NEXT A:RETURN
' *** SET UP GRAPHING
A$="GRAPHING SETUP":GOSUB 14210:GOSUB 14130:LOCATE 3,1,0:GOSUB 14140:IF A$ <> " " THEN RETURN
0 GOSUB 14710
0 GOSUB 44050:ON ERROR GOTO 33950:A$="ADISGRPH.BAS":CHAIN A$,ALL:RETURN
CLS:COLOR SBX,SA$:PRINT "PLEASE WAIT":COLOR SA$,SBX:RETURN
'*** PLOT AFTER EACH I/O CYCLE
HOX=HKX-1:IF HKX=0 THEN HOX=HMX-5 ' HOX & HKX point to data in array
IF HIX<HMX-9 THEN LINE (HIX+5,0)-(HIX+10,184),0,BF
FOR HHX= 0 TO GLX-G1X
IF GZ(HHX,0)=0 THEN 45300
H1X=HDX(HOX,HHX):H2X=HDX(HKX,HHX)
GOSUB 46350
IF GSX=0 OR SC=0 THEN 45200 ELSE IF SCX(HHX+G1X-1)=-1 THEN 45200
H1X=HSX(HOX,HHX):H2X=HSX(HKX,HHX)
GOSUB 46350
GOSUB 46450
NEXT HHX

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0 HIX=HIX+1:IF HIX>HM%-5 THEN HIX=0
20 RETURN
00 '***PLOT ARRAYS
0 IF HJX=0 THEN HPX=HKX ELSE HPX=HM%-5
20 FOR HHX= 0 TO GLX-G1X:IF GZ(HHX,0)=0 THEN 46220
0 HOX=HKX:REM HOX=PTR TO DATA IN HDX()
0 FOR HIX=0 TO HPX
5 HOX=HOX+1:IF HOX>HPX THEN HOX=0
0 H2X=HDH(HOX,HHX):GOSUB 46350:H1X=H2X
0 NEXT HIX:AA$=INKEY$:IF AA$<> "" THEN RETURN
0 IF GSX=0 OR SC=0 THEN 46150 ELSE IF SCX(HHX+G1X-1)=-1 THEN 46150
0 HOX=HKX
0 FOR HIX=0 TO HPX
5 HOX=HOX+1:IF HOX>HPX THEN HOX=0
0 H2X=HSX(HOX,HHX):GOSUB 46350:H1X=H2X
0 NEXT HIX:AA$=INKEY$:IF AA$<> "" THEN RETURN
0 GOSUB 46450
0 NEXT HHX:
0 IF HJX=0 THEN HIX=HPX+1 ELSE HIX=0
0 RETURN
0 IF H1>H9 OR H1<H0 THEN RETURN
0 H1X=184-(H1-H0)/(H9-H0)*184
0 LINE (5,H1X)-(HIX+5,H1X),GZ(HHX,3)
0 RETURN
0 IF HIX=0 THEN PSET((HIX+5),H2X),GZ(HHX,3):RETURN
0 LINE ((HIX+4),H1X)-((HIX+5),H2X),GZ(HHX,3)
0 RETURN
0 REM PLOT ALARMS
0 IF GAX<>1 OR SH<>1 THEN RETURN
0 H9=GZ(HHX,1):H0=GZ(HHX,2)
0 H1=SH(HHX+G1X-1):GOSUB 46300
0 H1=SL(HHX+G1X-1):GOSUB 46300
0 RETURN
0 '*** PRINT ERROR MESSAGE
0 CLS:BEEP:PRINT "Graphing was not set up in the Setup mode"
0 PRINT "Press any key to continue":BEEP
0 GS=INKEY$:IF GS="" THEN 46530
0 RETURN
0 ' *** SET INITIAL VALUE FOR LAST PLOT TIME
0 IF GEX=0 THEN HZ=TIMER-GH:HSFX=0:RETURN
0 HTT$=TIMES
0 HHH=VAL(MID$(HTT$,1,2))
0 HMM=VAL(MID$(HTT$,4,2))
0 HSS=VAL(MID$(HTT$,7,2))
0 ON GEX GOSUB 46690,46720,46750,46800
0 HST=TIMER:HZ=HST + HS - GH:HSFX=1:RETURN
0 '£ seconds till minute
0 HS=60-HSS:IF HS=60 THEN HS=0
0 RETURN
0 '£ seconds till hour
0 HS=(3600-(HMM*60)-HSS):IF HS=3600 THEN HS=0
0 RETURN
0 '£ seconds till 12 hrs
0 IF HMM>12 THEN HHG=HHH-12 ELSE HHG=HHH
0 HS=(43200!-(HHG*3600)-(HMM*60)-HSS)
0 IF HS=43200! THEN HS=0 ELSE IF HS<0 THEN HS=HS+43200!
0 RETURN
0 '£ seconds till 24 hrs
0 HS=(86400!-(HHH*3600)-(HMM*60)-HSS)
0 IF HS=86400! THEN HS=0
0 RETURN
0 REM ***PICTURE DISPLAY***
0 ARX=8:IF PM=0 OR PP=0 THEN CLS:PRINT "Picture mode not set up.":GOSUB 13310:AA$="" :RETURN
0 IF AP<>1 AND AP<>2 THEN CLS:PRINT "Color/Graphics adaptor not installed. Can't display picture":GOSUB 13310:AA$="" :RETURN
0 SCREEN PM,0:IF PM=1 THEN COLOR PC,PG

```

```

22 AL=LEN(SH$):IF LEN(SL$)>AL THEN AL=LEN(SL$)
24 AL=AL+1
26 ON ERROR GOTO 33950:A=0:DEF SEG=(&HB800):BLOAD PB$,A:DEF SEG:IF A>0 THEN RETURN
30 ON ERROR GOTO 0
40 IF AKX = 0 THEN AA$="":GOTO 47100
45 IF SNX<0 THEN 47070
50 FOR A=0 TO SNX:IF PTX(A)<1 THEN 47065
44 IF PM=1 AND SH=1 THEN IF AHX(A)>0 THEN COLOR PG,PC
46 LOCATE PVX(A),PHX(A),0:ON PTX(A) GOSUB 47210,47220,47230,47240
40 IF PM=1 THEN COLOR PC,PG
45 NEXT A
0 FOR A=0 TO SMX:IF PSX(A)<=0 THEN 47090
4 LOCATE PUX(A),PGX(A),0:PRINT SPACES$(AL);:LOCATE PUX(A),PGX(A),0:IF ALX(A)=1 AND PM=1 THEN COLOR SAX+16,SBX
6 IF AVX(A)=0 THEN PRINT SL$;
0 IF AVX(A)=1 THEN PRINT SH$;
0 NEXT A
0 GOSUB 10010
0 IF AA$="" THEN 47050
0 SCREEN 0,1:WIDTH 80:COLOR SAX,SBX:RETURN
0 IF SRX(A)<20 THEN PRINT USING SF$;AV(A); ELSE PRINT USING "####.##";AV(A);
5 RETURN
0 IF SM=0 THEN RETURN
4 IF SRX(A)<20 THEN PRINT USING SF$;AH(A); ELSE PRINT USING "####.##";AH(A);
6 RETURN
0 IF SM=0 THEN RETURN
4 IF SRX(A)<20 THEN PRINT USING SF$;AL(A); ELSE PRINT USING "####.##";AL(A);
6 RETURN
0 IF SB=0 THEN RETURN
4 IF SRX(A)<20 THEN PRINT USING SF$;AA(A); ELSE PRINT USING "####.##";AA(A);
6 RETURN
0 REM ***NO DISPLAY***
0 ARX=9:A$ = "NO DISPLAY":AA$="":GOSUB 14520:LOCATE 24,35,0:PRINT SPACES$(44);
0 GOSUB 10010
0 IF AA$="" THEN 48100
0 RETURN
0 REM ***SETUP***
0 ON ERROR GOTO 0:ANX = 0:AMX = ANX + 15: IF SNX < AMX THEN AMX = SNX
0 HKX=-1:APX = 0:AOX = APX + 15: IF SMX < AOX THEN AOX = SMX
0 REM SET UP TIME INTERVALS
0 GOSUB 11000:AR$ = TI$:AHX = VAL ( MID$ (TI$,14,2)): GOSUB 10510: REM AHX=HR FROM WHICH AQ IS TIMED
0 IF SQ$ < > "" THEN AX$ = SQ$:AY = AW: GOSUB 10710:AX = SQX: GOSUB 50210:AQ = AY
0 AJ=AW:AH=0
0 AKX = 0:RETURN
0 REM ADD 'START TIME'
0 IF AX = 0 THEN RETURN
0 IF AX = 1 THEN AY = ( VAL ( MID$ (TI$,17,2)) + 1) * 60: RETURN
0 IF AX = 2 THEN AY = 3600: RETURN
0 IF AX = 3 AND AHX < 12 THEN AY = (12 - AHX) * 3600: RETURN
0 IF AX = 3 THEN AY = (24 - AHX) * 3600: RETURN
0 AX = AHX:A$ = MID$ (TI$,23,1): IF A$ < > "H" AND AX = 12 THEN AX = 0
0 IF A$ = "P" THEN AY = (12 - AX) * 3600: RETURN
0 AY = (24 - AX) * 3600
0 RETURN
0 REM ADISPLAY V3.54 06-24-87

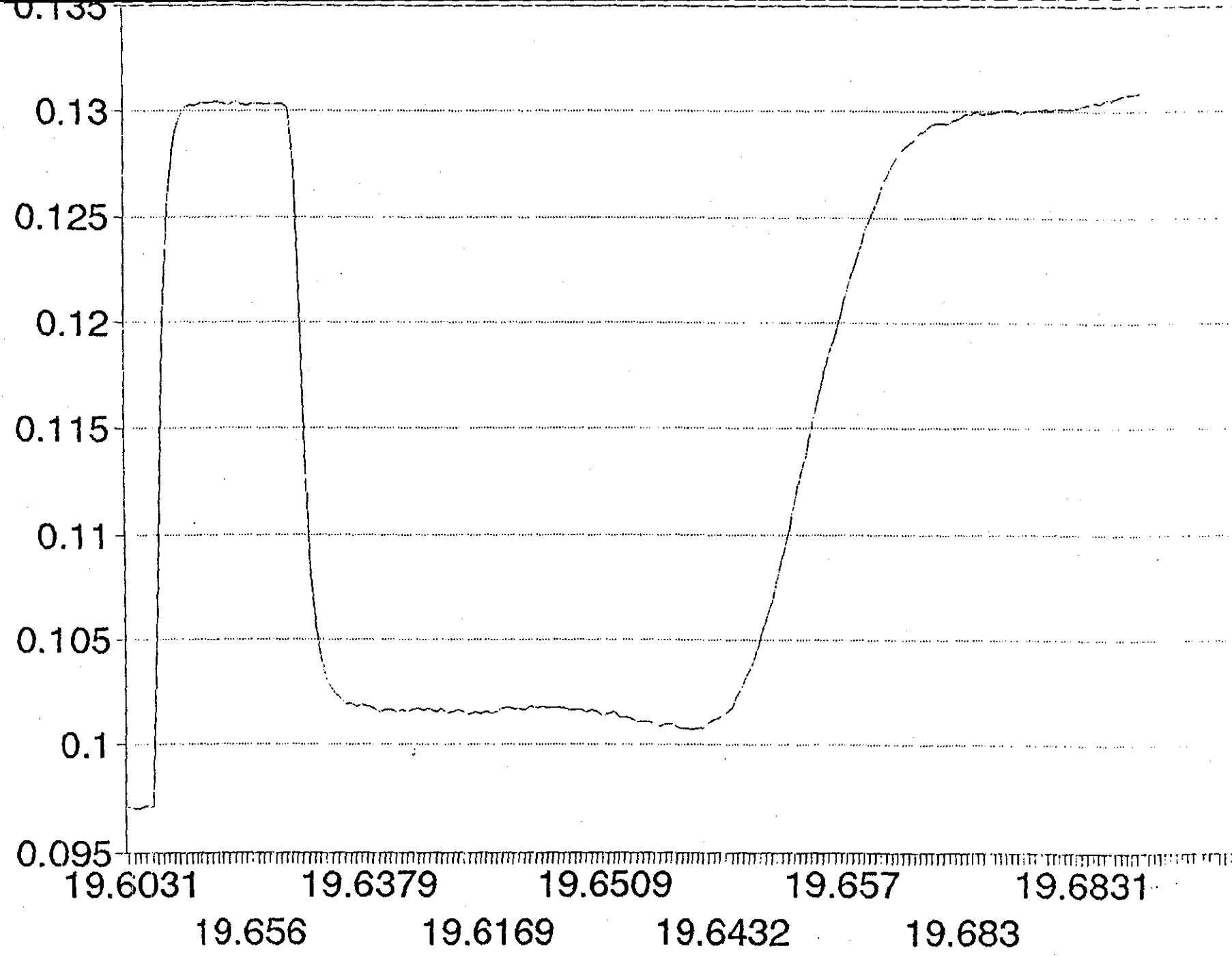
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## APPENDIX III

### COMPUTER DATA ANALYSIS USING QUATTRO SOFTWARE



-186-



# DATALOG

log interv	1	3	channels	12	digital I/O	31		
Year	Month	Day	Hour	Minute	Second	1/100 sec.	Present: c	
							1	2
91	8	19	11	11	32	79	19.6031	19.9127
91	8	19	11	11	35	70	19.5994	19.9177
91	8	19	11	11	38	77	19.5769	19.9153
91	8	19	11	17	31	67	19.6084	19.9570
91	8	19	11	17	34	80	19.6449	19.9728
91	8	19	11	17	37	77	19.6319	19.9495
91	8	19	11	17	40	7	19.6227	19.9730
91	8	19	11	17	42	38	19.6461	19.9706
91	8	19	11	17	45	29	19.6339	19.9756
91	8	19	11	17	47	60	19.6269	19.9530
91	8	19	11	17	49	91	19.6467	19.9453
91	8	19	11	17	52	87	19.6620	19.9623
91	8	19	11	17	55	18	19.6383	19.9524
91	8	19	11	17	57	49	19.6611	19.9717
91	8	19	11	18	0	40	19.6471	19.9630
91	8	19	11	18	2	70	19.6308	19.9604
91	8	19	11	18	5	1	19.6430	19.9744
91	8	19	11	18	7	92	19.6467	19.9694
91	8	19	11	18	10	23	19.6501	19.9453
91	8	19	11	18	12	54	19.6500	19.9502
91	8	19	11	18	15	45	19.6408	19.9669
91	8	19	11	18	17	75	19.6560	19.9580
91	8	19	11	18	20	6	19.6496	19.9568
91	8	19	11	18	22	97	19.6410	19.9638
91	8	19	11	18	25	28	19.6535	19.9503

91	8	19	11	18	27	58	19.6653	19.9449
91	8	19	11	18	30	55	19.6254	19.9620
91	8	19	11	18	32	86	19.6534	19.9606
91	8	19	11	18	35	16	19.6791	19.9638
91	8	19	11	18	38	8	19.6369	19.9528
91	8	19	11	18	40	38	19.6284	19.9788
91	8	19	11	18	42	69	19.6627	19.9664
91	8	19	11	18	45	60	19.6269	19.9324
91	8	19	11	18	47	91	19.6378	19.9520
91	8	19	11	18	50	21	19.6615	19.9566
91	8	19	11	18	53	13	19.6394	19.9397
91	8	19	11	18	55	43	19.6526	19.9805
91	8	19	11	18	57	74	19.6378	19.9605
91	8	19	11	19	0	65	19.6234	19.9703
91	8	19	11	19	2	96	19.6586	19.9796
91	8	19	11	19	5	26	19.6474	19.9477
91	8	19	11	19	8	17	19.6651	19.9723
91	8	19	11	19	10	48	19.6379	19.9451
91	8	19	11	19	12	79	19.6348	19.9523
91	8	19	11	19	15	75	19.6486	19.9524
91	8	19	11	19	18	6	19.6278	19.9592
91	8	19	11	19	20	37	19.6378	19.9570
91	8	19	11	19	23	28	19.6491	19.9581
91	8	19	11	19	25	59	19.6557	19.9577
91	8	19	11	19	27	89	19.6388	19.9460
91	8	19	11	19	30	80	19.6366	19.9559
91	8	19	11	19	33	11	19.6377	19.9536
91	8	19	11	19	35	42	19.6524	19.9562
91	8	19	11	19	37	72	19.6569	19.9658

91	8	19	11	19	40	64	19.6656	19.9503
91	8	19	11	19	42	94	19.6449	19.9763
91	8	19	11	19	45	25	19.6510	19.9478
91	8	19	11	19	48	16	19.6659	19.9334
91	8	19	11	19	50	47	19.6534	19.9571
91	8	19	11	19	52	77	19.6385	19.9560
91	8	19	11	19	55	74	19.6357	19.9602
91	8	19	11	19	58	5	19.6392	19.9447
91	8	19	11	20	0	35	19.6413	19.9347
91	8	19	11	20	3	27	19.6169	19.9484
91	8	19	11	20	5	57	19.6277	19.9470
91	8	19	11	20	7	88	19.6244	19.9679
91	8	19	11	20	10	79	19.6524	19.9596
91	8	19	11	20	13	10	19.6710	19.9522
91	8	19	11	20	15	40	19.6588	19.9643
91	8	19	11	20	18	31	19.6279	19.9455
91	8	19	11	20	20	62	19.6355	19.9668
91	8	19	11	20	22	93	19.6432	19.9624
91	8	19	11	20	25	84	19.6271	19.9620
91	8	19	11	20	28	15	19.6389	19.9738
91	8	19	11	20	30	45	19.6283	19.9528
91	8	19	11	20	33	36	19.6353	19.9442
91	8	19	11	20	35	67	19.6359	19.9604
91	8	19	11	20	37	98	19.6332	19.9542
91	8	19	11	20	40	89	19.6315	19.9559
91	8	19	11	20	43	20	19.6543	19.9477
91	8	19	11	20	45	45	19.6444	19.9550
91	8	19	11	20	48	41	19.6380	19.9263
91	8	19	11	20	50	72	19.6329	19.9384

91	8	19	11	20	53	3	19.6214	19.9718
91	8	19	11	20	55	94	19.6509	19.9408
91	8	19	11	20	58	25	19.6672	19.9382
91	8	19	11	21	0	55	19.6485	19.9281
91	8	19	11	21	3	46	19.6543	19.9425
91	8	19	11	21	5	77	19.6460	19.9291
91	8	19	11	21	8	8	19.6188	19.9312
91	8	19	11	21	10	99	19.6378	19.9450
91	8	19	11	21	13	30	19.6413	19.9157
91	8	19	11	21	15	60	19.6496	19.9412
91	8	19	11	21	18	51	19.6390	19.9324
91	8	19	11	21	20	82	19.6615	19.9186
91	8	19	11	21	23	13	19.6513	19.9274
91	8	19	11	21	26	4	19.6438	19.9372
91	8	19	11	21	28	34	19.6255	19.9293
91	8	19	11	21	30	65	19.6279	19.9541
91	8	19	11	21	33	56	19.6606	19.9419
91	8	19	11	21	35	87	19.6425	19.9583
91	8	19	11	21	38	18	19.6404	19.9476
91	8	19	11	21	41	9	19.6453	19.9508
91	8	19	11	21	43	39	19.6406	19.9271
91	8	19	11	21	45	70	19.6556	19.9421
91	8	19	11	21	48	61	19.6432	19.9556
91	8	19	11	21	50	92	19.6552	19.9693
91	8	19	11	21	53	23	19.6528	19.9427
91	8	19	11	21	56	19	19.6366	19.9576
91	8	19	11	21	58	50	19.6284	19.9580
91	8	19	11	22	0	81	19.6563	19.9635
91	8	19	11	22	3	11	19.6491	19.9529

91	8	19	11	22	6	2	19.6262	19.9334
91	8	19	11	22	8	33	19.6458	19.9789
91	8	19	11	22	10	64	19.6547	19.9585
91	8	19	11	22	13	60	19.6487	19.9542
91	8	19	11	22	15	91	19.6560	19.9701
91	8	19	11	22	18	22	19.6570	19.9711
91	8	19	11	22	21	13	19.6796	19.9694
91	8	19	11	22	23	43	19.6494	19.9445
91	8	19	11	22	25	74	19.6572	19.9557
91	8	19	11	22	28	65	19.6737	19.9774
91	8	19	11	22	30	96	19.6673	19.9711
91	8	19	11	22	33	27	19.6589	19.9747
91	8	19	11	22	36	18	19.6569	19.9693
91	8	19	11	22	38	48	19.6447	19.9709
91	8	19	11	22	40	79	19.6570	19.9727
91	8	19	11	22	43	76	19.6689	19.9743
91	8	19	11	22	46	6	19.6603	19.9537
91	8	19	11	22	48	37	19.6458	19.9547
91	8	19	11	22	51	28	19.6722	19.9603
91	8	19	11	22	53	59	19.6624	19.9575
91	8	19	11	22	55	90	19.6701	19.9531
91	8	19	11	22	58	86	19.6863	19.9779
91	8	19	11	23	1	17	19.6593	19.9700
91	8	19	11	23	3	48	19.6662	19.9492
91	8	19	11	23	6	39	19.6516	19.9795
91	8	19	11	23	8	69	19.6356	19.9600
91	8	19	11	23	11	0	19.6682	19.9736
91	8	19	11	23	13	91	19.6671	19.9621
91	8	19	11	23	16	22	19.6597	19.9582

91	8	19	11	23	18	53	19.6664	19.9752
91	8	19	11	23	21	44	19.6626	19.9784
91	8	19	11	23	23	74	19.6628	19.9838
91	8	19	11	23	26	5	19.6856	19.9772
91	8	19	11	23	28	96	19.6731	19.9906
91	8	19	11	23	31	27	19.6619	19.9932
91	8	19	11	23	33	57	19.6830	19.9867
91	8	19	11	23	36	49	19.6784	19.9700
91	8	19	11	23	38	79	19.6782	19.9663
91	8	19	11	23	41	10	19.6643	19.9697
91	8	19	11	23	44	7	19.6722	19.9793
91	8	19	11	23	46	37	19.6750	19.9855
91	8	19	11	23	48	68	19.6601	19.9827
91	8	19	11	23	51	59	19.6914	19.9795
91	8	19	11	23	53	90	19.6516	19.9778
91	8	19	11	23	56	20	19.6529	19.9997
91	8	19	11	23	59	17	19.6722	20.0018
91	8	19	11	24	1	48	19.6839	19.9840
91	8	19	11	24	3	78	19.6857	19.9790
91	8	19	11	24	6	69	19.6721	19.9792
91	8	19	11	24	9	0	19.6778	19.9987
91	8	19	11	24	11	31	19.6629	19.9821
91	8	19	11	24	14	22	19.6925	19.9892
91	8	19	11	24	16	53	19.6846	19.9917
91	8	19	11	24	18	83	19.6639	19.9676
91	8	19	11	24	21	74	19.6804	19.9841
91	8	19	11	24	24	5	19.6605	19.9676
91	8	19	11	24	26	36	19.6831	20.0161
91	8	19	11	24	28	67	19.6761	19.9867

91	8	19	11	24	31	58	19.6571	19.9867
91	8	19	11	24	33	88	19.6837	19.9925
91	8	19	11	24	36	19	19.6822	19.9755
91	8	19	11	24	39	16	19.6813	19.9901
91	8	19	11	24	41	46	19.6933	19.9849
91	8	19	11	24	43	71	19.6877	19.9759
91	8	19	11	24	46	63	19.6918	19.9903
91	8	19	11	24	48	93	19.7113	19.9942
91	8	19	11	24	51	24	19.6878	19.9949
91	8	19	11	24	54	15	19.6736	19.9756
91	8	19	11	24	56	46	19.6693	19.9834



Maximum			Minimum			Average:		
3	1	2	3	1	2	3	1	2
0.0971	19.6031	19.9127	0.0971	19.6031	19.9127	0.0971	19.60	19.91
0.0970	19.6031	19.9177	0.0971	19.5994	19.9127	0.0970	19.60	19.92
0.0971	19.5994	19.9177	0.0971	19.5769	19.9153	0.0970	19.59	19.92
0.0971	19.6084	19.9570	0.0971	19.6084	19.9570	0.0971	19.61	19.96
0.0971	19.6449	19.9728	0.0971	19.6084	19.9570	0.0971	19.63	19.96
0.1082	19.6449	19.9728	0.1082	19.6319	19.9495	0.0971	19.64	19.96
0.1205	19.6319	19.9730	0.1205	19.6227	19.9495	0.1082	19.63	19.96
0.1260	19.6461	19.9730	0.1260	19.6227	19.9706	0.1205	19.63	19.97
0.1288	19.6461	19.9756	0.1288	19.6339	19.9706	0.1260	19.64	19.97
0.1297	19.6339	19.9756	0.1297	19.6269	19.9530	0.1288	19.63	19.96
0.1302	19.6467	19.9530	0.1302	19.6269	19.9453	0.1297	19.64	19.95
0.1303	19.6620	19.9623	0.1303	19.6467	19.9453	0.1302	19.65	19.95
0.1303	19.6620	19.9623	0.1303	19.6383	19.9524	0.1303	19.65	19.96
0.1304	19.6611	19.9717	0.1304	19.6383	19.9524	0.1303	19.65	19.96
0.1304	19.6611	19.9717	0.1304	19.6471	19.9630	0.1304	19.65	19.97
0.1304	19.6471	19.9630	0.1304	19.6308	19.9604	0.1304	19.64	19.96
0.1305	19.6430	19.9744	0.1305	19.6308	19.9604	0.1304	19.64	19.97
0.1304	19.6467	19.9744	0.1305	19.6430	19.9694	0.1304	19.64	19.97
0.1303	19.6501	19.9694	0.1304	19.6467	19.9453	0.1303	19.65	19.96
0.1305	19.6501	19.9502	0.1305	19.6500	19.9453	0.1303	19.65	19.95
0.1303	19.6500	19.9669	0.1305	19.6408	19.9502	0.1303	19.65	19.96
0.1303	19.6560	19.9669	0.1303	19.6408	19.9580	0.1303	19.65	19.96
0.1304	19.6560	19.9580	0.1304	19.6496	19.9568	0.1303	19.65	19.96
0.1303	19.6496	19.9638	0.1304	19.6410	19.9568	0.1303	19.65	19.96
0.1303	19.6535	19.9638	0.1303	19.6410	19.9503	0.1303	19.65	19.96

0.1303	19.6653	19.9503	0.1303	19.6535	19.9449	0.1303	19.66	19.95
0.1304	19.6653	19.9620	0.1304	19.6254	19.9449	0.1303	19.65	19.95
0.1303	19.6534	19.9620	0.1304	19.6254	19.9606	0.1303	19.64	19.96
0.1302	19.6791	19.9638	0.1303	19.6534	19.9606	0.1302	19.67	19.96
0.1272	19.6791	19.9638	0.1302	19.6369	19.9528	0.1272	19.66	19.96
0.1214	19.6369	19.9788	0.1272	19.6284	19.9528	0.1214	19.63	19.97
0.1147	19.6627	19.9788	0.1214	19.6284	19.9664	0.1147	19.65	19.97
0.1084	19.6627	19.9664	0.1147	19.6269	19.9324	0.1084	19.64	19.95
0.1057	19.6378	19.9520	0.1084	19.6269	19.9324	0.1057	19.63	19.94
0.1042	19.6615	19.9566	0.1057	19.6378	19.9520	0.1042	19.65	19.95
0.1030	19.6615	19.9566	0.1042	19.6394	19.9397	0.1030	19.65	19.95
0.1026	19.6526	19.9805	0.1030	19.6394	19.9397	0.1026	19.65	19.96
0.1023	19.6526	19.9805	0.1026	19.6378	19.9605	0.1023	19.65	19.97
0.1019	19.6378	19.9703	0.1023	19.6234	19.9605	0.1019	19.63	19.97
0.1020	19.6586	19.9796	0.1020	19.6234	19.9703	0.1019	19.64	19.97
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0.1019	19.6651	19.9723	0.1019	19.6474	19.9477	0.1018	19.66	19.96
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0.1016	19.6557	19.9577	0.1016	19.6388	19.9460	0.1016	19.65	19.95
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0.1018	19.6389	19.9738	0.1019	19.6283	19.9528	0.1018	19.63	19.96
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0.1008	19.6279	19.9541	0.1009	19.6255	19.9293	0.1008	19.63	19.94
0.1008	19.6606	19.9541	0.1008	19.6279	19.9419	0.1008	19.64	19.95
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0.1036	19.6563	19.9635	0.1036	19.6284	19.9580	0.1029	19.64	19.96
0.1041	19.6563	19.9635	0.1041	19.6491	19.9529	0.1036	19.65	19.96

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0.1061	19.6458	19.9789	0.1061	19.6262	19.9334	0.1052	19.64	19.96
0.1069	19.6547	19.9789	0.1069	19.6458	19.9585	0.1061	19.65	19.97
0.1081	19.6547	19.9585	0.1081	19.6487	19.9542	0.1069	19.65	19.96
0.1092	19.6560	19.9701	0.1092	19.6487	19.9542	0.1081	19.65	19.96
0.1102	19.6570	19.9711	0.1102	19.6560	19.9701	0.1092	19.66	19.97
0.1116	19.6796	19.9711	0.1116	19.6570	19.9694	0.1102	19.67	19.97
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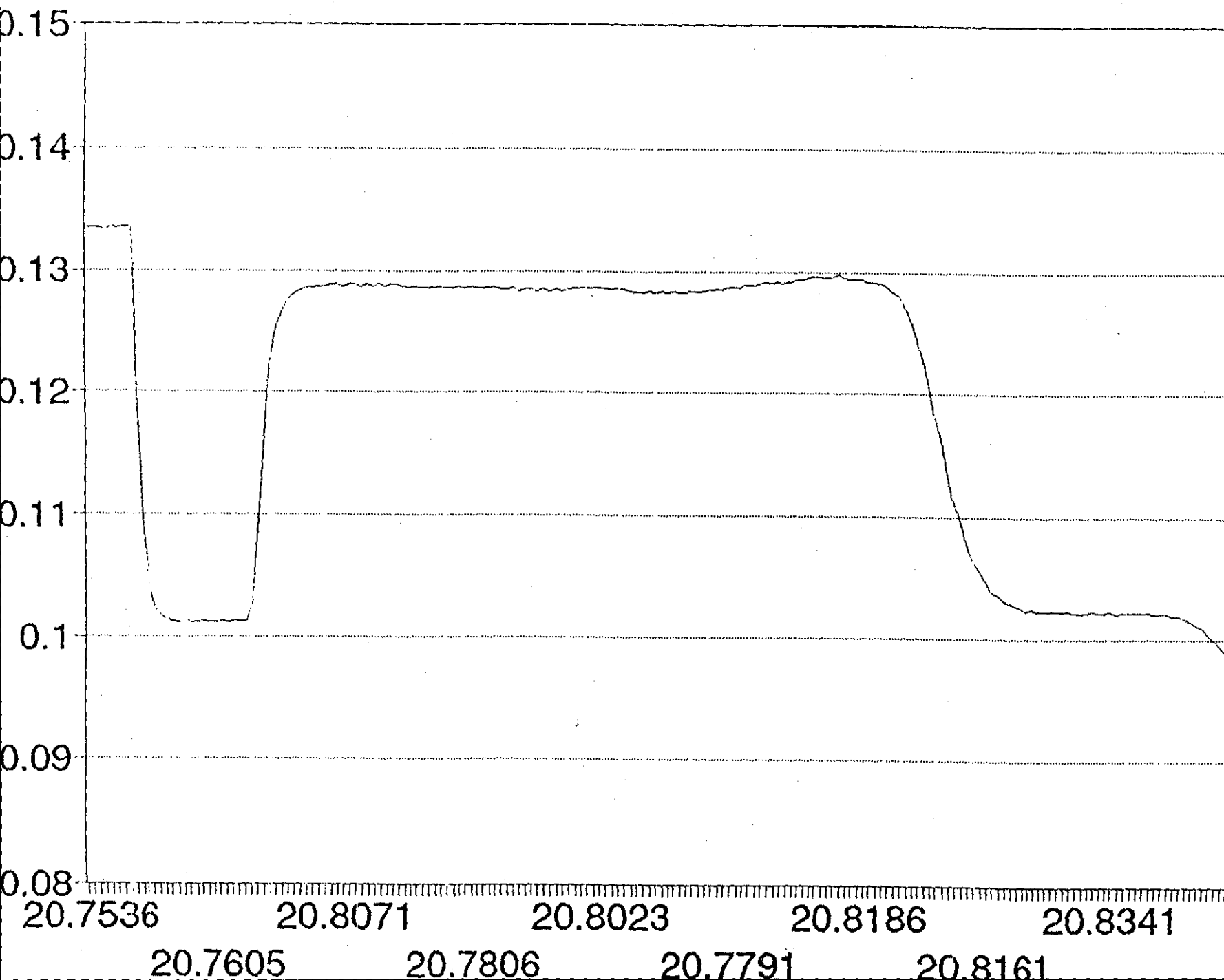
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Year	Month	Day	Hour	Minute	Second	1/100 sec. Present: c
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91	8	19	11	17	49	91 19.6467 19.9453
91	8	19	11	17	52	87 19.662 19.9623
91	8	19	11	17	55	18 19.6383 19.9524
91	8	19	11	17	57	49 19.6611 19.9717
91	8	19	11	18	0	40 19.6471 19.963
91	8	19	11	18	2	70 19.6308 19.9604
91	8	19	11	18	5	1 19.643 19.9744
91	8	19	11	18	7	92 19.6467 19.9694
91	8	19	11	18	10	23 19.6501 19.9453
91	8	19	11	18	12	54 19.65 19.9502
91	8	19	11	18	15	45 19.6408 19.9669
91	8	19	11	18	17	75 19.656 19.958
91	8	19	11	18	20	6 19.6496 19.9568
91	8	19	11	18	22	97 19.641 19.9638
91	8	19	11	18	25	28 19.6535 19.9503
91	8	19	11	18	27	58 19.6653 19.9449

91	8	19	11	18	30	55	19.6254	19.962
91	8	19	11	18	32	86	19.6534	19.9606
91	8	19	11	18	35	16	19.6791	19.9638
91	8	19	11	18	38	8	19.6369	19.9528
91	8	19	11	18	40	38	19.6284	19.9788
91	8	19	11	18	42	69	19.6627	19.9664
91	8	19	11	18	45	60	19.6269	19.9324
91	8	19	11	18	47	91	19.6378	19.952
91	8	19	11	18	50	21	19.6615	19.9566
91	8	19	11	18	53	13	19.6394	19.9397
91	8	19	11	18	55	43	19.6526	19.9805
91	8	19	11	18	57	74	19.6378	19.9605
91	8	19	11	19	0	65	19.6234	19.9703
91	8	19	11	19	2	96	19.6586	19.9796
91	8	19	11	19	5	26	19.6474	19.9477
91	8	19	11	19	8	17	19.6651	19.9723
91	8	19	11	19	10	48	19.6379	19.9451
91	8	19	11	19	12	79	19.6348	19.9523
91	8	19	11	19	15	75	19.6486	19.9524
91	8	19	11	19	18	6	19.6278	19.9592
91	8	19	11	19	20	37	19.6378	19.957
91	8	19	11	19	23	28	19.6491	19.9581
91	8	19	11	19	25	59	19.6557	19.9577
91	8	19	11	19	27	89	19.6388	19.946
91	8	19	11	19	30	80	19.6366	19.9559
91	8	19	11	19	33	11	19.6377	19.9536
91	8	19	11	19	35	42	19.6524	19.9562
91	8	19	11	19	37	72	19.6569	19.9658
91	8	19	11	19	40	64	19.6656	19.9503



91	8	19	11	19	42	94	19.6449	19.9763
91	8	19	11	19	45	25	19.651	19.9478
91	8	19	11	19	48	16	19.6659	19.9334
91	8	19	11	19	50	47	19.6534	19.9571
91	8	19	11	19	52	77	19.6385	19.956
91	8	19	11	19	55	74	19.6357	19.9602
91	8	19	11	19	58	5	19.6392	19.9447
91	8	19	11	20	0	35	19.6413	19.9347
91	8	19	11	20	3	27	19.6169	19.9484
91	8	19	11	20	5	57	19.6277	19.947
91	8	19	11	20	7	88	19.6244	19.9679
91	8	19	11	20	10	79	19.6524	19.9596
91	8	19	11	20	13	10	19.671	19.9522
91	8	19	11	20	15	40	19.6588	19.9643
91	8	19	11	20	18	31	19.6279	19.9455
91	8	19	11	20	20	62	19.6355	19.9668
91	8	19	11	20	22	93	19.6432	19.9624
91	8	19	11	20	25	84	19.6271	19.962
91	8	19	11	20	28	15	19.6389	19.9738
91	8	19	11	20	30	45	19.6283	19.9528
91	8	19	11	20	33	36	19.6353	19.9442
91	8	19	11	20	35	67	19.6359	19.9604
91	8	19	11	20	37	98	19.6332	19.9542
91	8	19	11	20	40	89	19.6315	19.9559
91	8	19	11	20	43	20	19.6543	19.9477
91	8	19	11	20	45	45	19.6444	19.955
91	8	19	11	20	48	41	19.638	19.9263
91	8	19	11	20	50	72	19.6329	19.9384
91	8	19	11	20	53	3	19.6214	19.9718

91	8	19	11	20	55	94	19.6509	19.9408
91	8	19	11	20	58	25	19.6672	19.9382
91	8	19	11	21	0	55	19.6485	19.9281
91	8	19	11	21	3	46	19.6543	19.9425
91	8	19	11	21	5	77	19.646	19.9291
91	8	19	11	21	8	8	19.6188	19.9312
91	8	19	11	21	10	99	19.6378	19.945
91	8	19	11	21	13	30	19.6413	19.9157
91	8	19	11	21	15	60	19.6496	19.9412
91	8	19	11	21	18	51	19.639	19.9324
91	8	19	11	21	20	82	19.6615	19.9186
91	8	19	11	21	23	13	19.6513	19.9274
91	8	19	11	21	26	4	19.6438	19.9372
91	8	19	11	21	28	34	19.6255	19.9293
91	8	19	11	21	30	65	19.6279	19.9541
91	8	19	11	21	33	56	19.6606	19.9419
91	8	19	11	21	35	87	19.6425	19.9583
91	8	19	11	21	38	18	19.6404	19.9476
91	8	19	11	21	41	9	19.6453	19.9508
91	8	19	11	21	43	39	19.6406	19.9271
91	8	19	11	21	45	70	19.6556	19.9421
91	8	19	11	21	48	61	19.6432	19.9556
91	8	19	11	21	50	92	19.6552	19.9693
91	8	19	11	21	53	23	19.6528	19.9427
91	8	19	11	21	56	19	19.6366	19.9576
91	8	19	11	21	58	50	19.6284	19.958
91	8	19	11	22	0	81	19.6563	19.9635
91	8	19	11	22	3	11	19.6491	19.9529
91	8	19	11	22	6	2	19.6262	19.9334

91	8	19	11	22	8	33	19.6458	19.9789
91	8	19	11	22	10	64	19.6547	19.9585
91	8	19	11	22	13	60	19.6487	19.9542
91	8	19	11	22	15	91	19.656	19.9701
91	8	19	11	22	18	22	19.657	19.9711
91	8	19	11	22	21	13	19.6796	19.9694
91	8	19	11	22	23	43	19.6494	19.9445
91	8	19	11	22	25	74	19.6572	19.9557
91	8	19	11	22	28	65	19.6737	19.9774
91	8	19	11	22	30	96	19.6673	19.9711
91	8	19	11	22	33	27	19.6589	19.9747
91	8	19	11	22	36	18	19.6569	19.9693
91	8	19	11	22	38	48	19.6447	19.9709
91	8	19	11	22	40	79	19.657	19.9727
91	8	19	11	22	43	76	19.6689	19.9743
91	8	19	11	22	46	6	19.6603	19.9537
91	8	19	11	22	48	37	19.6458	19.9547
91	8	19	11	22	51	28	19.6722	19.9603
91	8	19	11	22	53	59	19.6624	19.9575
91	8	19	11	22	55	90	19.6701	19.9531
91	8	19	11	22	58	86	19.6863	19.9779
91	8	19	11	23	1	17	19.6593	19.97
91	8	19	11	23	3	48	19.6662	19.9492
91	8	19	11	23	6	39	19.6516	19.9795
91	8	19	11	23	8	69	19.6356	19.96
91	8	19	11	23	11	0	19.6682	19.9736
91	8	19	11	23	13	91	19.6671	19.9621
91	8	19	11	23	16	22	19.6597	19.9582
91	8	19	11	23	18	53	19.6664	19.9752

91	8	19	11	23	21	44	19.6626	19.9784
91	8	19	11	23	23	74	19.6628	19.9838
91	8	19	11	23	26	5	19.6856	19.9772
91	8	19	11	23	28	96	19.6731	19.9906
91	8	19	11	23	31	27	19.6619	19.9932
91	8	19	11	23	33	57	19.683	19.9867
91	8	19	11	23	36	49	19.6784	19.97
91	8	19	11	23	38	79	19.6782	19.9663
91	8	19	11	23	41	10	19.6643	19.9697
91	8	19	11	23	44	7	19.6722	19.9793
91	8	19	11	23	46	37	19.675	19.9855
91	8	19	11	23	48	68	19.6601	19.9827
91	8	19	11	23	51	59	19.6914	19.9795
91	8	19	11	23	53	90	19.6516	19.9778
91	8	19	11	23	56	20	19.6529	19.9997
91	8	19	11	23	59	17	19.6722	20.0018
91	8	19	11	24	1	48	19.6839	19.984
91	8	19	11	24	3	78	19.6857	19.979
91	8	19	11	24	6	69	19.6721	19.9792
91	8	19	11	24	9	0	19.6778	19.9987
91	8	19	11	24	11	31	19.6629	19.9821
91	8	19	11	24	14	22	19.6925	19.9892
91	8	19	11	24	16	53	19.6846	19.9917
91	8	19	11	24	18	83	19.6639	19.9676
91	8	19	11	24	21	74	19.6804	19.9841
91	8	19	11	24	24	5	19.6605	19.9676
91	8	19	11	24	26	36	19.6831	20.0161
91	8	19	11	24	28	67	19.6761	19.9867
91	8	19	11	24	31	58	19.6571	19.9867

91	8	19	11	24	33	88	19.6837	19.9925
91	8	19	11	24	36	19	19.6822	19.9755
91	8	19	11	24	39	16	19.6813	19.9901
91	8	19	11	24	41	46	19.6933	19.9849
91	8	19	11	24	43	71	19.6877	19.9759
91	8	19	11	24	46	63	19.6918	19.9903
91	8	19	11	24	48	93	19.7113	19.9942
91	8	19	11	24	51	24	19.6878	19.9949
91	8	19	11	24	54	15	19.6736	19.9756
91	8	19	11	24	56	46	19.6693	19.9834

Maximum			Minimum			Average:		
3	1	2	3	1	2	3	1	2
0.097105	19.6031	19.9127	0.097105	19.6031	19.9127	0.097105	19.6	19.91
0.096995	19.6031	19.9177	0.097105	19.5994	19.9127	0.096995	19.6	19.92
0.097053	19.5994	19.9177	0.097053	19.5769	19.9153	0.096995	19.59	19.92
0.097137	19.6084	19.957	0.097137	19.6084	19.957	0.097137	19.61	19.96
0.09706	19.6449	19.9728	0.097137	19.6084	19.957	0.09706	19.63	19.96
0.108207	19.6449	19.9728	0.108207	19.6319	19.9495	0.09706	19.64	19.96
0.120452	19.6319	19.973	0.120452	19.6227	19.9495	0.108207	19.63	19.96
0.126033	19.6461	19.973	0.126033	19.6227	19.9706	0.120452	19.63	19.97
0.128838	19.6461	19.9756	0.128838	19.6339	19.9706	0.126033	19.64	19.97
0.129653	19.6339	19.9756	0.129653	19.6269	19.953	0.128838	19.63	19.96
0.130225	19.6467	19.953	0.130225	19.6269	19.9453	0.129653	19.64	19.95
0.130285	19.662	19.9623	0.130285	19.6467	19.9453	0.130225	19.65	19.95
0.13026	19.662	19.9623	0.130285	19.6383	19.9524	0.13026	19.65	19.96
0.130421	19.6611	19.9717	0.130421	19.6383	19.9524	0.13026	19.65	19.96
0.130404	19.6611	19.9717	0.130421	19.6471	19.963	0.130404	19.65	19.97
0.130392	19.6471	19.963	0.130404	19.6308	19.9604	0.130392	19.64	19.96
0.130463	19.643	19.9744	0.130463	19.6308	19.9604	0.130392	19.64	19.97
0.130398	19.6467	19.9744	0.130463	19.643	19.9694	0.130398	19.64	19.97
0.13027	19.6501	19.9694	0.130398	19.6467	19.9453	0.13027	19.65	19.96
0.13047	19.6501	19.9502	0.13047	19.65	19.9453	0.13027	19.65	19.95
0.130332	19.65	19.9669	0.13047	19.6408	19.9502	0.130332	19.65	19.96
0.130262	19.656	19.9669	0.130332	19.6408	19.958	0.130262	19.65	19.96
0.13035	19.656	19.958	0.13035	19.6496	19.9568	0.130262	19.65	19.96
0.130339	19.6496	19.9638	0.13035	19.641	19.9568	0.130339	19.65	19.96
0.130325	19.6535	19.9638	0.130339	19.641	19.9503	0.130325	19.65	19.96
0.130337	19.6653	19.9503	0.130337	19.6535	19.9449	0.130325	19.66	19.95

0.13036	19.6653	19.962	0.13036	19.6254	19.9449	0.130337	19.65	19.95
0.130341	19.6534	19.962	0.13036	19.6254	19.9606	0.130341	19.64	19.96
0.130219	19.6791	19.9638	0.130341	19.6534	19.9606	0.130219	19.67	19.96
0.127234	19.6791	19.9638	0.130219	19.6369	19.9528	0.127234	19.66	19.96
0.121384	19.6369	19.9788	0.127234	19.6284	19.9528	0.121384	19.63	19.97
0.114747	19.6627	19.9788	0.121384	19.6284	19.9664	0.114747	19.65	19.97
0.108412	19.6627	19.9664	0.114747	19.6269	19.9324	0.108412	19.64	19.95
0.105695	19.6378	19.952	0.108412	19.6269	19.9324	0.105695	19.63	19.94
0.104186	19.6615	19.9566	0.105695	19.6378	19.952	0.104186	19.65	19.95
0.103022	19.6615	19.9566	0.104186	19.6394	19.9397	0.103022	19.65	19.95
0.102606	19.6526	19.9805	0.103022	19.6394	19.9397	0.102606	19.65	19.96
0.102344	19.6526	19.9805	0.102606	19.6378	19.9605	0.102344	19.65	19.97
0.101918	19.6378	19.9703	0.102344	19.6234	19.9605	0.101918	19.63	19.97
0.10201	19.6586	19.9796	0.10201	19.6234	19.9703	0.101918	19.64	19.97
0.101831	19.6586	19.9796	0.10201	19.6474	19.9477	0.101831	19.65	19.96
0.101933	19.6651	19.9723	0.101933	19.6474	19.9477	0.101831	19.66	19.96
0.101854	19.6651	19.9723	0.101933	19.6379	19.9451	0.101854	19.65	19.96
0.10177	19.6379	19.9523	0.101854	19.6348	19.9451	0.10177	19.64	19.95
0.101614	19.6486	19.9524	0.10177	19.6348	19.9523	0.101614	19.64	19.95
0.101641	19.6486	19.9592	0.101614	19.6278	19.9524	0.101614	19.64	19.96
0.101672	19.6378	19.9592	0.101672	19.6278	19.957	0.101641	19.63	19.96
0.101619	19.6491	19.9581	0.101672	19.6378	19.957	0.101619	19.64	19.96
0.10164	19.6557	19.9581	0.10164	19.6491	19.9577	0.101619	19.65	19.96
0.101585	19.6557	19.9577	0.10164	19.6388	19.946	0.101585	19.65	19.95
0.101687	19.6388	19.9559	0.101687	19.6366	19.946	0.101585	19.64	19.95
0.101715	19.6377	19.9559	0.101715	19.6366	19.9536	0.101687	19.64	19.95
0.101686	19.6524	19.9562	0.101715	19.6377	19.9536	0.101686	19.65	19.95
0.101695	19.6569	19.9658	0.101695	19.6524	19.9562	0.101686	19.65	19.96
0.101604	19.6656	19.9658	0.101695	19.6569	19.9503	0.101604	19.66	19.96

0.101711	19.6656	19.9763	0.101711	19.6449	19.9503	0.101604	19.66	19.96
0.101539	19.651	19.9763	0.101711	19.6449	19.9478	0.101539	19.65	19.96
0.101568	19.6659	19.9478	0.101568	19.651	19.9334	0.101539	19.66	19.94
0.101644	19.6659	19.9571	0.101644	19.6534	19.9334	0.101568	19.66	19.95
0.101559	19.6534	19.9571	0.101644	19.6385	19.956	0.101559	19.65	19.96
0.10145	19.6385	19.9602	0.101559	19.6357	19.956	0.10145	19.64	19.96
0.101559	19.6392	19.9602	0.101559	19.6357	19.9447	0.10145	19.64	19.95
0.101472	19.6413	19.9447	0.101559	19.6392	19.9347	0.101472	19.64	19.94
0.10162	19.6413	19.9484	0.10162	19.6169	19.9347	0.101472	19.63	19.94
0.10154	19.6277	19.9484	0.10162	19.6169	19.947	0.10154	19.62	19.95
0.101578	19.6277	19.9679	0.101578	19.6244	19.947	0.10154	19.63	19.96
0.101701	19.6524	19.9679	0.101701	19.6244	19.9596	0.101578	19.64	19.96
0.101798	19.671	19.9596	0.101798	19.6524	19.9522	0.101701	19.66	19.96
0.10169	19.671	19.9643	0.101798	19.6588	19.9522	0.10169	19.66	19.96
0.10173	19.6588	19.9643	0.10173	19.6279	19.9455	0.10169	19.64	19.95
0.101675	19.6355	19.9668	0.10173	19.6279	19.9455	0.101675	19.63	19.96
0.101824	19.6432	19.9668	0.101824	19.6355	19.9624	0.101675	19.64	19.96
0.101797	19.6432	19.9624	0.101824	19.6271	19.962	0.101797	19.64	19.96
0.10188	19.6389	19.9738	0.10188	19.6271	19.962	0.101797	19.63	19.97
0.101814	19.6389	19.9738	0.10188	19.6283	19.9528	0.101814	19.63	19.96
0.101757	19.6353	19.9528	0.101814	19.6283	19.9442	0.101757	19.63	19.95
0.101773	19.6359	19.9604	0.101773	19.6353	19.9442	0.101757	19.64	19.95
0.101769	19.6359	19.9604	0.101773	19.6332	19.9542	0.101769	19.63	19.96
0.101629	19.6332	19.9559	0.101769	19.6315	19.9542	0.101629	19.63	19.96
0.101687	19.6543	19.9559	0.101687	19.6315	19.9477	0.101629	19.64	19.95
0.101654	19.6543	19.955	0.101687	19.6444	19.9477	0.101654	19.65	19.95
0.101614	19.6444	19.955	0.101654	19.638	19.9263	0.101614	19.64	19.94
0.10168	19.638	19.9384	0.10168	19.6329	19.9263	0.101614	19.64	19.93
0.10159	19.6329	19.9718	0.10168	19.6214	19.9384	0.10159	19.63	19.96



0.101364	19.6509	19.9718	0.10159	19.6214	19.9408	0.101364	19.64	19.96
0.101549	19.6672	19.9408	0.101549	19.6509	19.9382	0.101364	19.66	19.94
0.101594	19.6672	19.9382	0.101594	19.6485	19.9281	0.101549	19.66	19.93
0.101344	19.6543	19.9425	0.101594	19.6485	19.9281	0.101344	19.65	19.94
0.101343	19.6543	19.9425	0.101344	19.646	19.9291	0.101343	19.65	19.94
0.101241	19.646	19.9312	0.101343	19.6188	19.9291	0.101241	19.63	19.93
0.101136	19.6378	19.945	0.101241	19.6188	19.9312	0.101136	19.63	19.94
0.101138	19.6413	19.945	0.101138	19.6378	19.9157	0.101136	19.64	19.93
0.101105	19.6496	19.9412	0.101138	19.6413	19.9157	0.101105	19.65	19.93
0.100966	19.6496	19.9412	0.101105	19.639	19.9324	0.100966	19.64	19.94
0.100926	19.6615	19.9324	0.100966	19.639	19.9186	0.100926	19.65	19.93
0.100984	19.6615	19.9274	0.100984	19.6513	19.9186	0.100926	19.66	19.92
0.10097	19.6513	19.9372	0.100984	19.6438	19.9274	0.10097	19.65	19.93
0.100879	19.6438	19.9372	0.10097	19.6255	19.9293	0.100879	19.63	19.93
0.100805	19.6279	19.9541	0.100879	19.6255	19.9293	0.100805	19.63	19.94
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0.100701	19.6606	19.9583	0.100798	19.6425	19.9419	0.100701	19.65	19.95
0.100792	19.6425	19.9583	0.100792	19.6404	19.9476	0.100701	19.64	19.95
0.100783	19.6453	19.9508	0.100792	19.6404	19.9476	0.100783	19.64	19.95
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0.101206	19.6556	19.9421	0.101206	19.6406	19.9271	0.101036	19.65	19.93
0.101299	19.6556	19.9556	0.101299	19.6432	19.9421	0.101206	19.65	19.95
0.101508	19.6552	19.9693	0.101508	19.6432	19.9556	0.101299	19.65	19.96
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0.110197	19.657	19.9711	0.110197	19.656	19.9701	0.109234	19.66	19.97
0.111567	19.6796	19.9711	0.111567	19.657	19.9694	0.110197	19.67	19.97
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0.116353	19.6737	19.9774	0.116353	19.6673	19.9711	0.115313	19.67	19.97
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0.130687	1	1	1	1	1	1	1	1
0.130763	1	1	1	1	1	1	1	1
0.13081	1	1	1	1	1	1	1	1
0.130878	1	1	1	1	1	1	1	1
0.131033	1	1	1	1	1	1	1	1

