AN ANALYSIS OF THE DESIGN AND PRODUCTION OF INERT GAS ATMOSPHERES FOR INDUSTRIAL PROCESSES

© JOHN McLOUGHLIN

MASTER OF SCIENCE DEGREE
UNIVERSITY OF GLASGOW
DEPARTMENT OF MECHANICAL ENGINEERING

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ProQuest LLC.
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To all members of the Works Engineers Department I express my thanks as each had a role to play at some stage in the preparation of this thesis. In particular my colleagues John Fitchie and John Leckey who provided the moral support in the closing days of the work. I thank Neil Savage for his help during the experimental phases and Graham Pyper who gave me invaluable assistance in preparing the illustrations.

Finally, as so often, I thank my wife Irene for her patience.

DECLARATION

I declare that this Thesis has been prepared by myself.

During the research and preparation of the document I have referred to work by others in the field of inert atmospheres and oxygen enriched atmospheres.

The development of gaseous mixing models has been on the basis of well accepted techniques with supporting experimentation conducted by myself.

The analysis of the particular application on the autoclave and the development of the computer simulation model is my own original work.

SUMMARY

The analysis of the design and creation of inert atmospheres for industrial processes can be divided into three areas:-

(1) A general view of the need for an inert atmosphere and the behaviour of gases, vapours and solids in these atmospheres when under the influence of various temperatures and pressures with the accent towards the control of combustion hazards.

(2) How to design systems to create these inert atmospheres using simple computer modelling techniques.

(3) The review of a real situation using computer models and simulation to determine the optimum inert gas plant configuration to support a production programme.

Chapter 1 consists of a review of the data available on the quality of inert atmosphere required for the prevention and control of combustion, and the effectiveness of the various inert gases and agents in this role.

This data is usually for ambient temperatures and atmospheric pressure, but if modified atmospheres are encountered created by higher temperatures and pressures this data is no longer valid. This aspect is examined in Chapter 2 with one particular form of modified atmosphere classified as "Oxygen Enriched". These atmospheres can radically change the

combustion characteristics of materials by widening the combustion range and reducing the ignition energy and temperature required to initiate combustion. Under these conditions it is necessary to create higher concentrations of inert gas than suggested by the data in Chapter 1.

To create the desired quality of inert atmosphere it is necessary to purge a vessel with inert gas and this aspect is examined in Chapter 3. One of the important factors in this process is the need for high levels of gas movement induced by the inert gas entering the vessel or by mechanical means within the vessel such as a fan. The basic model as frequently applied in industry in the design of purging systems is reviewed and its limitations identified. An improvement is suggested using a simple computer modelling technique in which a large complex volume is broken down into smaller lumped parameter volumes. These sub-volumes are linked together by gas movement across the boundaries. It is suggested that this approach can produce a closer response to that of the real plant and also provides a greater insight to the mechanisms within the vessel.

In Chapter 4 the theories postulated in Chapter 3 are put to the test through experimentation on two pieces of apparatus . One is a simple clear volume and the other is an approximate model of an autoclave. The results have shown that high mean gas velocities and high turbulence levels are important for fast and effective mixing. The basic model as currently applied provides a poor approximation to the response of a system particularly in the later stages of a purge cycle where it tends to over estimate the inert gas required to create a specific

atmosphere. The experiments have shown that by breaking down complex volumes into multiple lumped parameter systems greater accuracy can be attained particularly when the technique is linked to information on gas movement obtained through experiments.

Chapters 5 and 6 are devoted to the review of a real application for inert atmospheres which is an autoclave in the aerospace industry. Under certain circumstances there is the possibility of combustion within the vessel and the aim is to prevent this by creating an inert atmosphere.

There are many facets in the design of an inert gas plant in addition to the purging process such as how frequently will the demand for inert gas be imposed on the system, and what is the optimum size and configuration of the key components to support a production schedule ?

The approach was to develop a computer simulation model for the complete autoclave system based a procedural model for the production programme, a computational model for the processes and a purge model based on the knowledge gained through Chapters 3 and 4.

The combustion hazard was reviewed in the light of the data and information in Chapters 1 and 2 and the quality of atmosphere required for the processes was deduced.

The aim of the analysis was to design the optimum plant configuration to meet the process demand, but with the minimum capital and running cost. The procedure was to run the simulation model with various combinations of the key components such as

compressors, nitrogen plant etc. and obtain a set of equations relating cost to the size of these key components. Once this was achieved the equations were processed by multiple linear regression to derive a general equation. The optimum solution to this equation was obtained by applying linear programming techniques. The proposed solution was then run on the simulation model and proved to be successful.

This inert gas plant had originally been designed by manual steady state methods and using a basic simple purging model. On the basis of this design the cost of the inert gas plant was estimated at £500,000 with an annual running cost of £160,000. By determining the extent of the real combustion hazard in the light of this research and by applying the design techniques described in this thesis it has been possible to reduce these costs. The results have shown that by using the design produced through this work a saving of £60,000 (12%) can be made on the initial capital cost and a £50,000 (31%) on the annual running cost.

INTRODUCTION

The presence of oxygen during certain industrial processes is unacceptable as it can cause unwanted reactions and disrupt the intended process. These reactions can be between the oxygen and the process materials producing products of poor quality or the oxygen can combine with other factors and create a combustion hazard.

As 20.9% of air is atmospheric oxygen it is necessary to exclude it from processes where these problems are experienced, and this is achieved by the creation of an envelope of inert gas around the process. The technique of creating this inert atmosphere is known as purging or inerting. Gases such as nitrogen, helium, argon and carbon dioxide or manufactured agents such as halongenated hydrocarbons are often used in the role.

The aim of this work will be to review the application of inert atmospheres for combustion control in sealed vessels and to suggest design procedures that determine accurately the amount of gas required to achieve the aims of the purging process in the most economical manner.

The current design methods are inaccurate with the general tendency to over estimate the quantity of inert gas required to obtain a specific atmosphere. As the cost of inert gas can be high it is important to accurately determine the amount of gas required to purge a vessel.

A practical application designed by current methods will be reviewed. This is a nitrogen production plant which was designed to prevent combustion within a high temperature, high pressure vessel known as an Autoclave. The inert gas plant will be redesigned using the methods suggested in this thesis and the results compared.

As the estimated cost of the inert plant based on the initial design was £500,000 with an annual running cost of £160,000 there is ample justification in attempting to increase the accuracy of the design and avoid an over-sized and inefficient plant.

The format adopted within the each chapter is to introduce the particular subject and then deal with the various aspects in detail with conclusions at the end of the chapter. There is in addition a chapter at the end of the thesis (Chapter 7) which contains a general discussion of the work and summarises the conclusions reached.

CHAPTER I

INERT ATMOSPHERES

1.0 INTRODUCTION

The process of inerting or purging is defined as the creation of an atmosphere consisting substantially of inert gas to prevent unwanted reactions which may occur if oxygen is present.

The unwanted reactions fall into two general groups defined in this study as the **Process Group** and **Combustion Group** with the work concentrating on this latter group.

This chapter will review the data available on the quality of inert atmosphere required for various applications at atmospheric pressure and ambient temperature in the Combustion Group and the performance of the inert gases in this role of combustion control.

1.1 PROCESS GROUP

The aim of inerting in this application is to prevent reactions between process materials and oxygen which if allowed to take place would produce a product of unacceptable quality or disrupt the desired process reaction. Examples of such situations arise in food processing and during the manufacture of semi-conductors for the electronics industry.

The inert gases normally considered in these applications are nitrogen or argon. The former is the preferred selection owing to the availability and lower cost as it can be readily extracted from the atmosphere. Nitrogen is not totally inert as it can

form nitrides at high temperatures which can affect product quality. For this reason it is not used in furnaces or during certain welding operations. Here argon which remains stable at high temperatures is more suitable.

1.2 COMBUSTION GROUP

A frequently occurring application with a high demand for inert gas is in the prevention of combustion. The situation arises in a wide variety of industries from atomic power to petrochemical and food processing. The hazard takes the general form of a closed vessel with a fuel/oxidant mixture of gas, vapour or dust and a potential ignition source capable of imparting sufficient energy to create a reaction.

GAS	T(°K)	Cp(J/Kg°K)	k(W/m°K)
CO_2	300	871	0.0166
Nitrogen	300	1041	0.0262
Helium	255	5200	0.1357

Thermophysical Properties of Diluent Gases at Atmospheric Pressure

TABLE 1.1 [Ref: 1.0]

The creation of an inert atmosphere prevents the combustion reaction by one of two mechanisms.

The first is by creating a smothering effect in which the inert gas, if in sufficient quantity, provides a physical barrier to the effective interaction of the fuel/oxidant molecules in the mixture. The relative effectiveness of these gases correlates with their specific heat capacity and thermal conductivity. It

can be shown that carbon dioxide is more effective than nitrogen which in turn is more effective than helium. **[See: Table 1.1].**

The second mechanism is that in which the inert gas interferes with the combustion chemical reaction by having an anticatalytic effect. Gases that are capable of creating this effect fall within the group called halogenated hydrocarbons known as Halons.

The performance of the Halon gases depends on the number and type of halogen atoms in the molecule. The relative effectiveness of various atoms is as follows:-

Iodine > Bromide > Chlorine > Fluorine

[Ref: 2.1]

Owing to the toxicity of Iodine it is not used and so the agents available contain various combinations of the other three atoms. If an agent is to be suitable for inerting it must be volatile and those used are Halons 1001, 1211 and 1301 **[See: Table 1.2].**

HALON NO	NAME	FORMULAE	BOILING.PT.
1001	MB	CH_3Br	+4.5°C
1211	BCF	$CBrClF_2$	-4.0°C
1301	BTM	$CBrF_3$	-57.6°C

INERTING HALON AGENTS

TABLE 1.2

1.3 INERT ATMOSPHERE CONCENTRATIONS

The concentration of inert gas required varies with the particular application and it is important to set this level at its optimum value as it is uneconomical to create 100% inert atmospheres in every situation particularly when large volumes of gas are necessary.

The Process Group of applications permit only extremely low levels of oxygen in the atmosphere and typically the aim will be to reduce the total oxygen content to 0.5 - 2.0%.

There are applications where it is necessary to reduce levels further. These arise in the semi-conductor industry where all the air must be removed and the purity of the inert gas is controlled to ensure that the oxygen content is in the range 0.5 - 4VPM.

When considering the Combustion Group there is greater flexibility available, depending on the application and two concentrations are of interest [Ref: 3.1].

(a) **The flame extinguishing concentration**

The minimum concentration in the air feeding a diffusion flame which will extinguish the flame.

(b) **The inerting concentration:**

The minimum concentration which will prevent the propagation of a premixed flame in a flammable atmosphere.

The essential difference between these two concentration levels is that the former is designed to extinguish the flames of a fire while the latter aims to create an atmosphere that will prevent an explosion. A higher level of inert gas is required for explosion prevention than that required for extinguishing.

1.4 FLAME EXTINGUISHING CONCENTRATION

A study of flame extinguishing concentrations has been carried out by Hirst and Booth [Ref: 2.0] in which they used a cup burner apparatus to test the effectiveness of various gases in a flame extinguishing role using liquid fuels such as n-Heptane, Acetone etc.

The results are tabulated in **Table 1.3** and indicate the amount of gas[1] or agent required to extinguish a flame created by the combustion of a selection of liquid fuels. From the results it can be observed that it would be possible to extinguish any of the flames by introducing nitrogen and increasing its concentration to 40% in this nitrogen/air atmosphere.

[1] The amount of extinguishing gas or agent is in addition to any similar gas that may be present in air. Typically in the case of nitrogen the percentage concentrations given in **Table 1.3** are in addition to the nitrogen already present in air (Approx. 70%).

FUEL	HALON 1001	HALON 1211	HALON 1301	CO_2	N_2
	Flame Extinguishing Concentration % by Volume				
n-Heptane	5.1	3.8	3.5	20.5	30.2
Acetone	4.6	3.8	3.5	18.3	29.2
Benzene	4.4	2.9	2.9	20.1	29.7
Diethylether	5.2	4.4	3.9	22.2	35.0
Ethanol	5.3	4.5	3.9	21.8	33.3
n-Hexane		3.7	3.3	19.5	31.0
Methanol	7.8	8.2	7.3	26.2	39.0
n-Pentane	5.0	3.7	3.3	19.0	31.5

Flame Extinguishing Concentrations in Air

(Ambient Temperature and Pressure)

Table 1.3 (Ref: 2.0)

The results in **Table 1.3** were obtained from experiments in the laboratory and these have been compared to full scale tests. It has been found that the laboratory flame was more difficult to extinguish than those experienced in the full scale tests.

The results tabulated in **Table 1.3** clearly illustrate that the Halons are more effective than the inert gases such as nitrogen and carbon dioxide. With the combustion of methanol a Halon 1211 concentration of 8.2% will extinguish the flame, whereas a nitrogen concentration of 39% is required. This fact is important if it is necessary to extinguish a fire quickly as it obviously takes considerably less time to create an inert atmosphere consisting of 8.2% Halon 1211 than 39% nitrogen.

1.5 INERTING CONCENTRATION

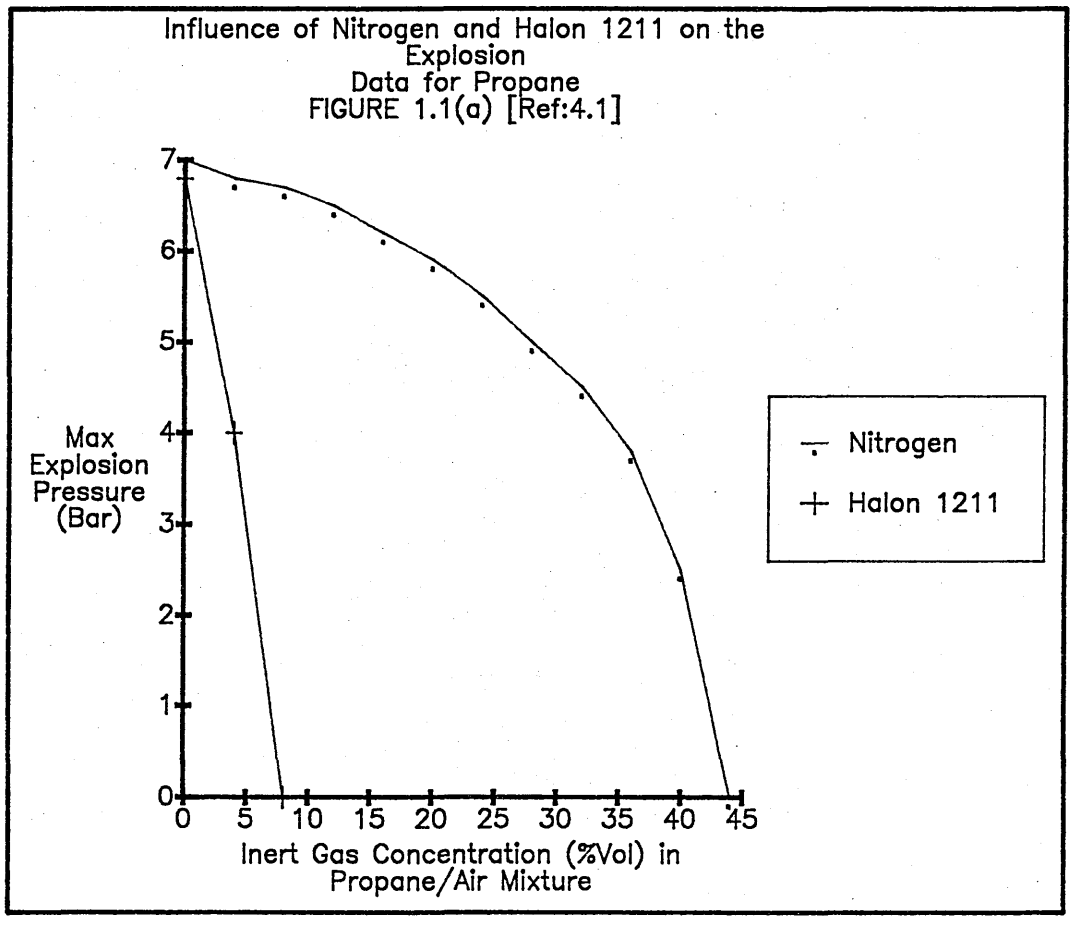
Work has been carried out by Bartkneckt [Ref: 4.0] on the performance of the inert gases and agents in the explosion prevention role. The effect of nitrogen and Halon 1211 on the explosive characteristic of propane is illustrated in **Figures 1.1** and **1.2.**

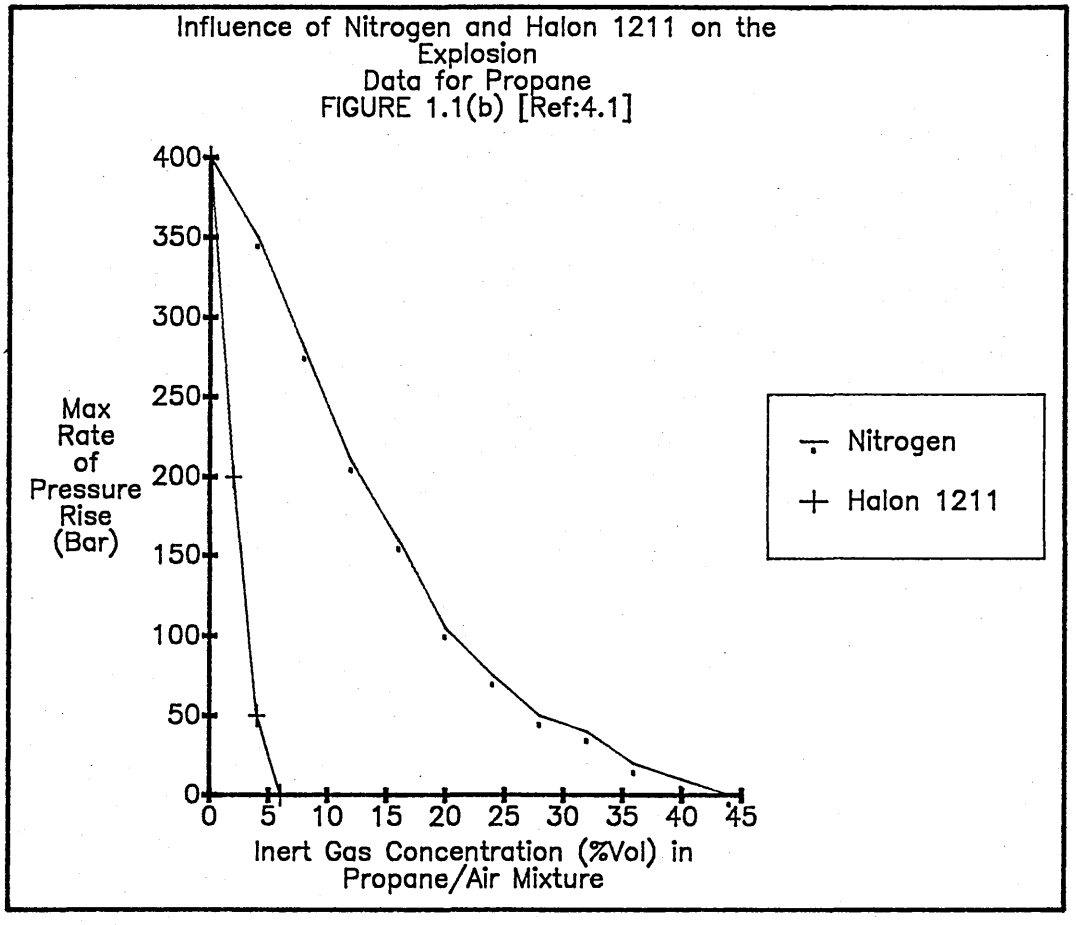
A measure of the violence of an explosion is the rate of pressure rise (dp/dt) **[See: Section 2.4.2]** **Figure 1.1** illustrates how the violence of a propane explosion can be reduced by the introduction of either nitrogen or Halon 1211.

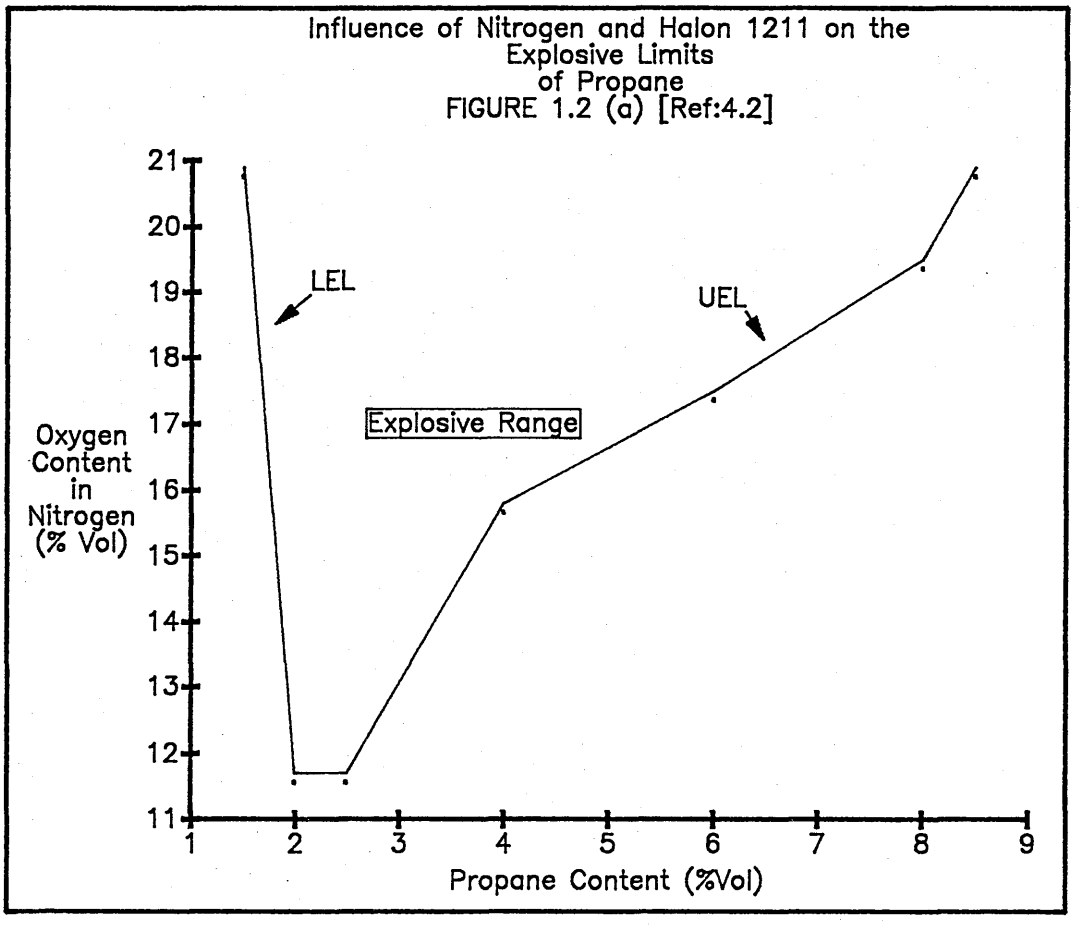
In explosion control Halon 1211 is considerably more effective than nitrogen, even with a small quantity of Halon 1211, reducing the oxygen content by a few percent there is a marked reduction in the violence of the explosion, and if the Halon content is above 7% in the propane/air mixture an explosion will be prevented. In order to prevent an explosion with nitrogen under the same conditions it is necessary to provide a nitrogen content in excess of 45% in this propane/air mixture. **[See Figure 1.1].**

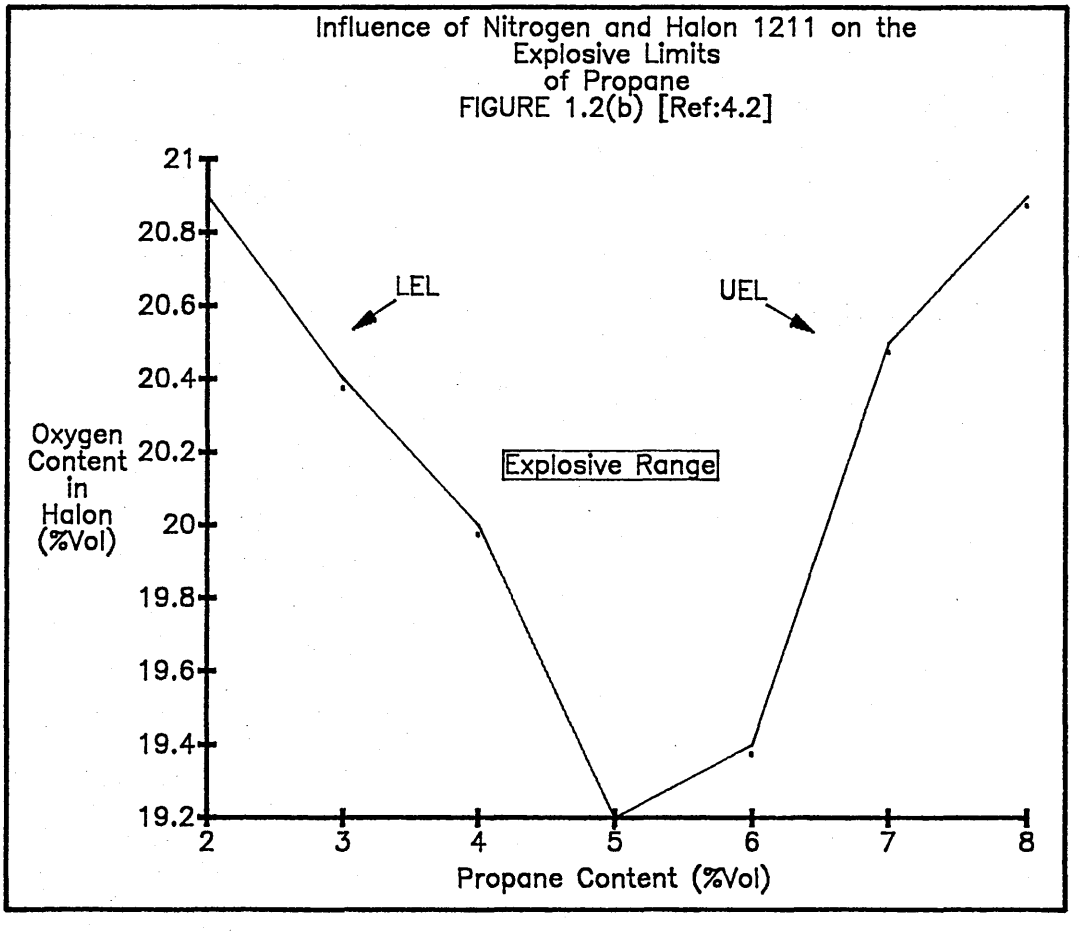
For an explosion to occur it is necessary for the fuel/oxidant mixture to be within the explosive range **[See:Section 2.4.3].** This explosive range is bounded by the upper explosive limit (UEL) and the lower explosive limit (LEL). The influence of Halon 1211 and nitrogen on the explosive limits of propane is illustrated in **Figure 1.2.** As the percentage of inert gas or agent increases the oxygen content reduces and the explosive limits narrow until eventually the mixture is no longer explosive.

The rate of change in the explosive range is dependent upon the inert gas, but it is important to realise that the upper and lower explosive limits may not


Influence of Nitrogen and Halon 1211 on the Explosion
Data for Propane
FIGURE 1.1(a) [Ref:4.1]
Max Explosion Pressure (Bar)
0
1
2
3
4
5
6
7
0 5 10 15 20 25 30 35 40 45
Inert Gas Concentration (%Vol) in Propane/Air Mixture
Nitrogen
Halon 1211


Influence of Nitrogen and Halon 1211 on the Explosion
Data for Propane
FIGURE 1.1(b) [Ref:4.1]
Max Rate of Pressure Rise (Bar)
0
50
100
150
200
250
300
350
400
0 5 10 15 20 25 30 35 40 45
Inert Gas Concentration (%Vol) in Propane/Air Mixture
Nitrogen
Halon 1211


Influence of Nitrogen and Halon 1211 on the
Explosive Limits
of Propane
FIGURE 1.2 (a) [Ref:4.2]
Oxygen Content in Nitrogen (% Vol)
21
20
19
18
17
16
15
14
13
12
11
LEL
UEL
Explosive Range
1
2
3
4
5
6
7
8
9
Propane Content (%Vol)


Influence of Nitrogen and Halon 1211 on the
Explosive Limits
of Propane
FIGURE 1.2(b) [Ref:4.2]
Oxygen Content in Halon (%Vol)
21
20.8
20.6
20.4
20.2
20
19.8
19.6
19.4
19.2
LEL
UEL
Explosive Range
2
3
4
5
6
7
8
Propane Content (%Vol)

change in the same manner. This is vividly illustrated in **Figure 1.2(a)** with a nitrogen/oxygen mixture. As the oxygen concentration is reduced due to the introduction of nitrogen the upper explosive limit changes reducing the explosive range whilst the lower explosive limit remains relatively constant. The result is that with a 2.25% propane content and sufficient nitrogen to reduce the oxygen level to 12% the oxygen/nitrogen/propane mixture is still in the explosive range. If the propane content is increased above 3% however, the mixture no longer constitutes an explosion hazard. Care should therefore be taken when dealing with this mixture near the lower explosive limit and low propane concentrations.

It should be noted that although the mixture with 2.25% propane and 12% oxygen is still in the explosive range the high percentage of nitrogen does have a beneficial effect as the violence of any explosion is considerably reduced as indicated by **Figure 1.1(b)**.

As an indication of the quality of inert atmosphere required to prevent explosions in combustible gases and dusts **Table 1.4** and **1.5** have been included. These tables indicate the maximum allowable oxygen concentration when inerting with nitrogen at ambient temperature and atmospheric pressure. They illustrate that contrary to popular opinion, and current design methods, it is not necessary in every case to create extremely low levels of oxygen concentration for combustion or explosion control at atmospheric pressure.

It is important to note however, that if the temperature or pressure is raised the flammable and explosive ranges widen particularly in the upper region and lower oxygen concentrations may be required and the data in Tables 1.4 and 1.5 may be no longer valid. This aspect of modified atmospheres will be examined in Chapter 2.

Flammable Gas or Vapour	Maximum Allowable O_2 Concentration (% Vol)
Methane	12.0
Hexane	12.0
Propylene	11.5
Benzene	11.2
Propane	11.0
Ethane	11.0
Ethylene	10.0
Coke Gas	7.0
Hydrogen	4.0

Maximum Allowable Oxygen Content for Inerting Flammable Gases and Vapours with Nitrogen.

Table 1.4 [Ref: 4.3]

DUST	MAXIMUM ALLOWABLE O_2 CONCENTRATION (% VOL)
Coal Dust	14.0
Cadium Laureate	14.0
Barium Stearate	13.0
Organic Pigment	12.0
Cadium Stearate	11.9
Calcium Stearate	11.8
Wood Dust	11.0
Resin Dust	10.0
Methyl Cellulose	10.0
Light Metal Dust	4 to 6

Maximum Allowable Oxygen Content for Inerting
Combustible Dusts with Nitrogen
(High-energy ignition source)

Table 1.5 (Ref: 4.4)

CHAPTER 2

OXYGEN ENRICHED ATMOSPHERES

2.0 INTRODUCTION

Chapter 1 provided a review of the type and quality of inert atmosphere for a variety of roles mainly in the control of combustion hazards. Most of the data available is for air at ambient temperature and atmospheric pressure. Combustion characteristics change in modified atmospheres where different temperatures and pressures are experienced.

This chapter will review the effect of oxygen enriched atmospheres on the combustion characteristics of materials in hyperbaric vessels. An oxygen enriched atmosphere is one in which the partial pressure of oxygen exceeds the normal value for atmospheric conditions which is 212 mbar. When air pressure is increased there is a corresponding increase in the. partial pressure of the oxygen even though the proportion of oxygen is not altered.

These conditions arise frequently during industrial processes and a particular application will be reviewed in Chapters 5 and 6 where oxygen enriched atmospheres combined with high temperatures are created within an Autoclave.

2.1 OXYGEN ENRICHED ATMOSPHERES

The commonly encountered problem with oxygen enriched atmospheres is accelerated combustion which is due to the combined effect of pressure, temperature and the composition of the atmosphere.

Not all atmospheres which fall within this oxygen enriched definition create an increased fire hazard

nor support the combustion of normally flammable materials. The results from tests with filter paper strips in a range of oxygen/nitrogen atmospheres at various pressures are shown in **Figure 2.1 [Ref: 5.1]**.

In these tests oxygen-nitrogen atmospheres were created and a range of experiments conducted with a variety of combinations of oxygen concentration and total pressure.

Filter paper strips were introduced and ignition was attempted with a resistance wire igniter. All the combinations that are above the lower dotted line are oxygen enriched atmospheres. The results on the graph fall within three zones described as complete combustion, incomplete combustion and non-combustion. There is a fourth case not shown as a zone on the graph called slight combustion.

The following is the definition of four cases:-

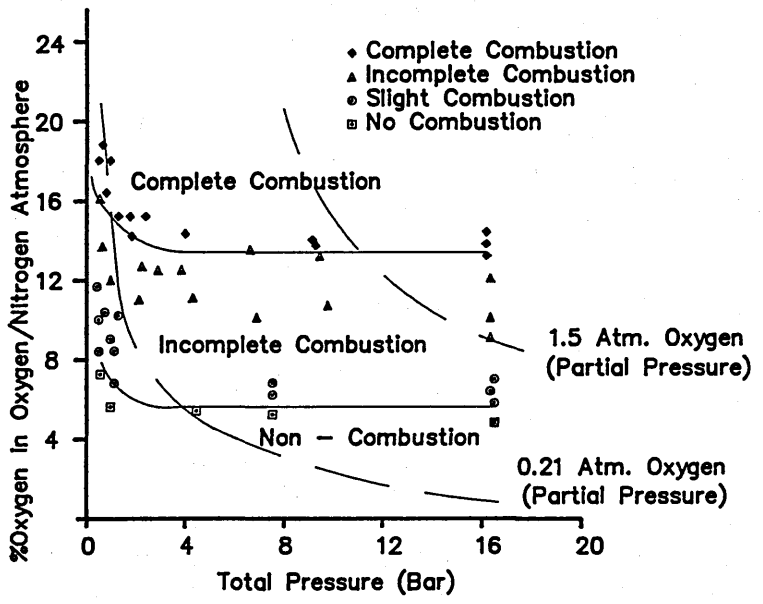
(1) **Complete Combustion -**
The filter paper strip burns completely

(2) **Incomplete Combustion -**
The filter paper strips burns for a length greater than one centimetre, but the flame extinguishes itself before the strip is completely consumed

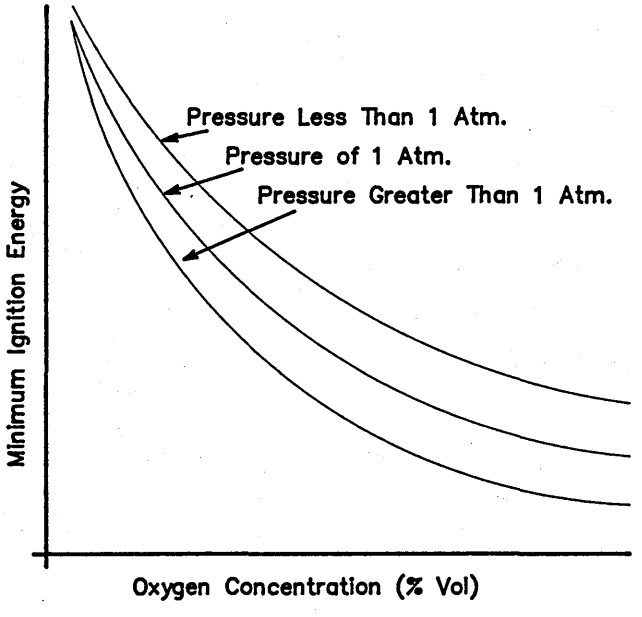
(3) **Slight Combustion -**
The filter paper strip flames or smokes, but does not burn more than one centimetre

(4) **Non Combustion - No ignition**

Take a specific case of an atmosphere at a total pressure of 12 bar, a gaseous mixture of 90% nitrogen and 4% oxygen. With an oxygen partial pressure of



Varying Degrees of Combustion in Oxygen Enriched Atmospheres
FIGURE 2.1 [Ref: 5.1]



Minimum Ignition Energy Behaviour of Combustibles
in Oxygen–Diluent Atmospheres at Different Pressures
FIGURE 2.2 [Ref: 6.1]

490 mbar the conditions fall within the definition of oxygen enriched. From inspection of **Figure 2.1** however, it can be observed that such an atmosphere will not support the combustion of paper.

2.2 IGNITION

To initiate the combustion process it is necessary to have an ignition source that is capable of imparting to the fuel and oxygen molecules sufficient energy for the chemical interaction to take place. The minimum ignition energy varies with the type of ignition source; the specific chemical nature and physical character of the combustible; and the composition, pressure and temperature of the atmosphere.

It is not possible to set down specific relationships between environmental characteristics and ignition energy which can be applied in all cases. The data must be determined experimentally for the particular application under investigation. The three environmental factors which influence ignition are the oxygen concentration, pressure and temperature. If one of these is considered a variable and the other two remain constant the general trend in the relationship with ignition is as follows:-

Oxygen Concentration:-

The minimum ignition energy varies inversely with the concentration of oxygen.

Pressure:-

The minimum ignition energy varies inversely with the change in pressure.

Temperature:-

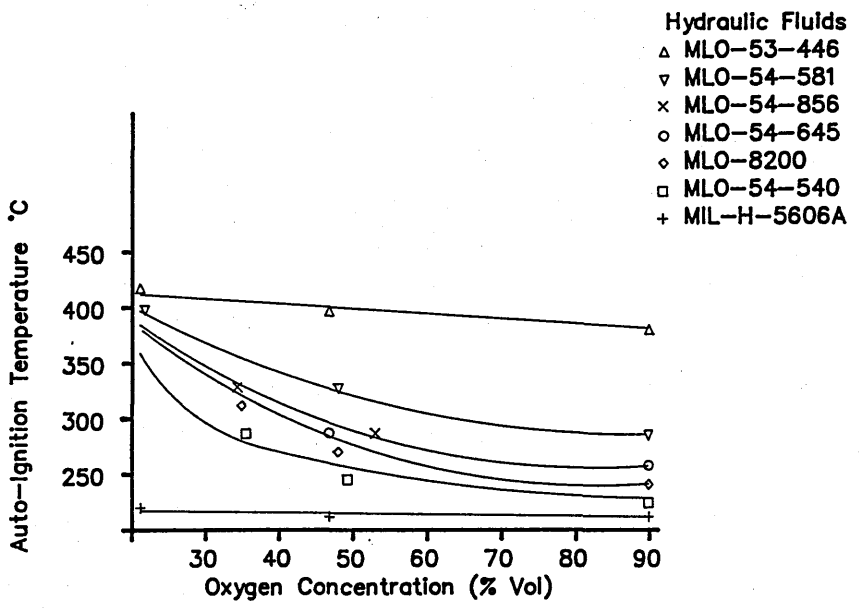
As the temperature of a system rises the ignition energy required reduces until at a sufficiently high temperature the mixture will ignite spontaneously. (Auto-ignition Temperature).

These first two relationships are graphically illustrated in **Figure 2.2 [Ref: 6.1].**

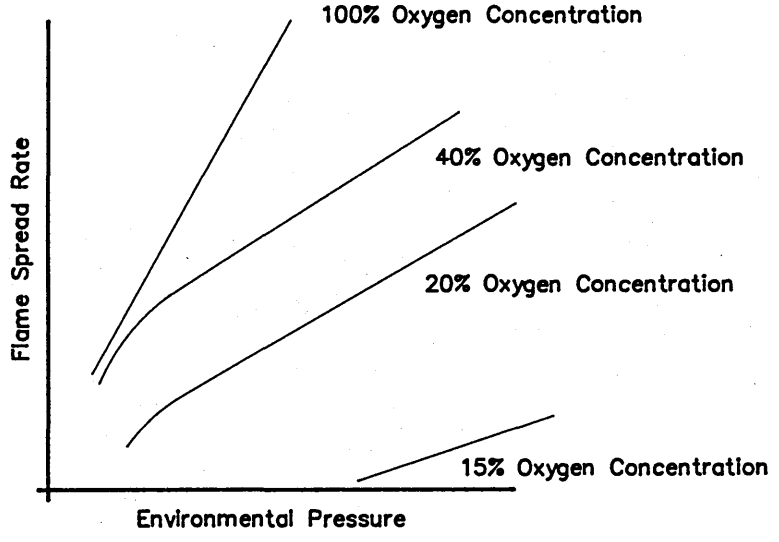
It is often the process materials that are examined closely to ensure that they do not provide a source of ignition, but there are other materials that can cause a problem. Hydraulic oil may be part of the control system and inadvertently enter the high pressure environment thus creating a hazard. The effect of a change in the partial pressure of oxygen on the auto-ignition temperature of hydraulic oil is clearly demonstrated in **Figure 2.3 [Ref: 5.2].** Data is not available on the combustion characteristic of hydraulic oils in various oxygen partial pressures due to increased pressure, but **Figure 2.3** illustrates the same effect. The total pressure remains constant, but the oxygen concentration varies thus affecting the partial pressure in the same way as increasing the total pressure.

Source of Ignition

(1)	Electrical Sources	Electrostatic Break (arc) sparks
(2)	Hot Surfaces	Friction sparks Heated wires Hot metal surfaces
(3)	Foreign Material	Illegal combustible material



Minimum Auto-Ignition Temperature of Seven Hydraulic Fluids
at Atmospheric Pressure in Various Oxygen/Nitrogen Atmospheres
FIGURE 2.3 [Ref: 5.2]



Effect of Atmospheric Oxygen and Environmental Pressure
on Flame Spread Rate
FIGURE 2.4 [Ref: 7.1]

2.3 COMBUSTION

Once the process of ignition has taken place it is necessary, if combustion is to continue, for a supply of fuel and oxygen molecules to chemically interact and this must be self sustained by the process.

The combustion of products in the oxygen enriched atmosphere will be considered in two broad groups.

(1) The combustion of liquids, vapours and gases.

(2) The combustion of solids.

2.3.1 Liquids, Vapours and Gases

When considering the combustion of liquids, vapours and gases there are two types of mixing - homogeneous and heterogeneous.

Homogeneous mixing will occur with vapours or gases and is the state when the combustible and oxidant are in intimate and uniform contact. A mixture in this state is considered flammable if the specific state lies between the limits of flammability for the particular atmosphere temperature and pressure. The effect of an increase in the total pressure, temperature or oxygen concentration is to broaden the flammability limits particularly in the upper region.

The more common mixing condition is that called heterogeneous and can be in the form of a single phase or multiphase system. A single phase system would be considered as one with gaseous fuel oxidant mixture, while liquid injected into an oxygen atmosphere would be considered as multiphase.

Single phase systems respond in a similar manner to that of homogeneous mixtures however, multiphase

systems require higher ignition energy to vaporise the fuel droplets to form heterogeneous single phase mixtures.

The type of flame normally encountered under these circumstances is called a molecular diffusion flame where the fuel and oxygen molecules diffuse to the flame front and chemically react in the form of combustion. In cases where there is gas movement with relatively high velocities then turbulent diffusion may predominate and in special cases a premixed flame may be established.

A molecular diffusion flame has a lower temperature than a turbulent diffusion flame or a premixed flame and it is easier to extinguish with lower concentrations of inert gas than required for the latter flame types.

Provided the fuel supply rate remains constant the burning rate is determined by the oxygen concentration.

Table 2.1 [Ref: 5.3] details the ignition and flammability properties of some combustible liquids and gases in air at atmospheric pressure. The following are the definitions of the headings in **Table 2.1 (Ref: 5.3)**

Flash Point:-
The minimum temperature of a liquid at which it gives off sufficient vapour to form an ignitable mixture with a gaseous oxidant near the surface under specified environmental conditions.

Minimum Ignition Temperature:-

The minimum temperature required to initiate self-sustaining combustion independently of any heated element. (Also known as Auto ignition temperature).

Minimum Ignition Energy:-

The minimum energy required to ignite a flammable mixture.

	Flash Point	Min. Ignit Temp	Min. Ignit Energy	Flammability LFL	UFL
	°C	°C	mj	%	%
		HYDROCARBON FUELS			
Methanegas	gas	537	0.30	5.0	15.0
Ethylene	gas	490	0.07	2.7	36.0
Propylene	gas	458	0.28	2.4	11.0
Gasoline (100/300)	-10	440		1.3	7.1
Kersoine	37.8	227		0.7	5.0
		SOLVENTS			
Methyl Alcohol	12.2	385	0.14	6.7	36.0
Ethyl Alcohol	12.8	365		3.3	19.0
Glycol	111.0	400		3.5	
Acetone	-17.8	465	1.15	2.6	13.0
Trichloroethane		458	2.37	6.2	16.0
Trichloroethylene	32.2	420	18.0	10.5	41.0
Carbon Tetrachloride		Non Flammable			
		MISCELLANEOUS COMBUSTIBLES			
Acetic Acid	40	465		5.4	
Carbon Monoxide	gas	609		12.5	74.0
Carbon Disulfide	-30	90	0.015	1.3	50.0
Hydrogen	gas	500	0.017	4.0	75.0

Ignition and Flammability Properties of Combustible Liquids and Gases in Air at Atmospheric Pressure

Table 2.1 [Ref: 5.3]

2.3.2 SOLIDS

The combustion of a solid requires that a portion of the heat of combustion is transferred back into the solid to cause its vaporisation providing more gaseous fuel to react with the oxygen.

The flammability of a solid is related to the partial pressure of the oxygen in the atmosphere, therefore an increase in the partial pressure may transfer a solid from a non-flammable classification to one considered flammable.

Table 2.2 [Ref: 5.4] details the results of tests on 4 materials in air at various pressures and illustrates clearly the reduction in ignition temperature with a rise in pressure.

		Ignition Temperature °C Total Pressure, Atmospheres			
MATERIAL	OXIDANT	1	2	3	4
Cotton Sheeting	Air	465	425	385	365
Rubber Sheeting	Air	480	395	375	370
Paper Drapes	Air	470	455	425	405
PVC	Air	+600	---	495	490

Minimum Hot Plate Ignition Temperatures of 4 Combustible Materials in Air at Various Total Pressures

TABLE 2.2 [Ref: 5.4]

There are many variables to be considered when reviewing the flammability of solids:-

(1) The specimen's physical characteristics.

(2) The ignition source.

(3) Orientation of the specimen.

(4) Environmental characteristics.

(5) Inerting dilutents.

(6) Contamination with more flammable materials.

A measure of the flammability of a solid is the flame spread rate which increases as the oxygen partial pressure increases. The variation in the flame spread rate with environmental pressure and oxygen concentration is illustrated in **Figure 2.4 [Ref: 7.1]**. The flame is normally a diffusion flame with the fuel and oxygen molecules diffusing to the flame front and there interacting to cause combustion.

Certain conditions can greatly increase flame spread rate apart from the changes in the oxygen partial pressure. Typically if a solid is contaminated with more flammable material such as oil, or if the surface of the solid consists of fine fibres as with fabrics. In the latter case the flame spread rate can rise to a value in the order of 500mm/sec. Similarly oily fabrics may ignite with a spark energy as small as 1/10000th of that for a clean sample. **[Ref: 5.0]**.

This is an important factor in the hazard analysis for any process. An illegal combustible material can be left within a vessel and subjected to environmental conditions that make it ignite. A hazard analysis considering only the ignition energy levels of the process materials may suggest the materials do not constitute a hazard under normal process conditions. If material such as an oil contaminated rag were left accidently the conditions could change radically. The rag may ignite and provide sufficiently high energy levels to create secondary ignition of the process materials.

2.4 EXPLOSIONS

2.4.1 Definition

Explosions are a particularly severe form of combustion which cover the range from that described as a deflagration with the definition of a rapid combustion without the generation of a shock wave, to a detonation, defined as an extremely rapid combustion resulting in the generation of a shock wave in the combustible mixture.

The combustible mixture in this case would consist of either a flammable gas/air or combustible dust/air mixture.

2.4.2 Pressure

There are two pressures of interest when considering explosions:-

(1) Maximum rate of pressure rise.

(2) Maximum explosion pressure.

The maximum rate of pressure rise is directly related to the flame spread rate and hence a measure of the violence of the explosion. This pressure rise varies between combustibles and is related to the volume of the vessel within which the explosion takes place. In general the maximum rate of pressure rise is characterised by the "Cubic Law" which is dealt with later. **[See: Section 2.4.4].**

The maximum explosive pressure is not related to the volume, but rather the substance involved and the mixture. For common flammable gases under normal conditions the maximum explosive pressure is in the range 7 to 10 bar. **[Ref: 4.6].**

A typical pressure characteristic of an explosion is illustrated in **Figure 2.5 [Ref: 4.7]**.

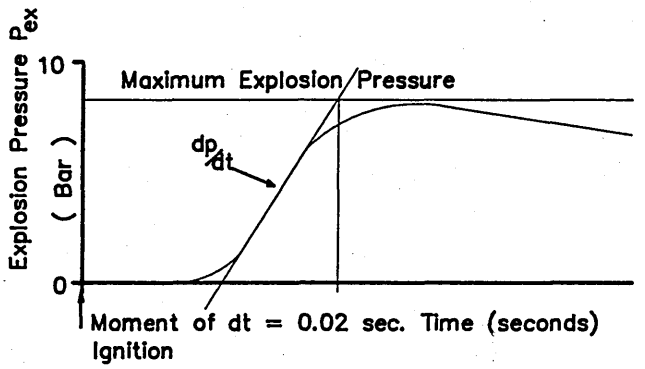
2.4.3 Explosion Limits

As in normal combustion for a mixture of gas, vapour, or dust to be flammable to the point of explosion the fuel/oxidant mixture and the environmental conditions must be such as to place the specific mixture within the explosive range. This range is bounded by the:-

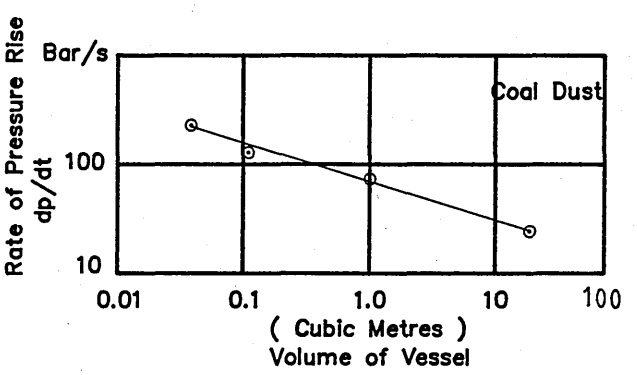
LEL - Lower Explosive Limit
UEL - Upper Explosive Limit

Table 2.3 [Ref: 4.8] gives the explosive limits for a number of flammable gases and vapours.

The situation is not so straight forward when considering combustible dusts because the mixing is no longer simply between gas molecules, but gas molecules and particles. To be explodable the particle size must be below a certain limit and in suspension within a certain concentration range.



The Typical Pressure Characteristic of an Explosion
FIGURE 2.5 [Ref: 4.7]



Validity of the Cubic Law for
Coal Dust Explosions
FIGURE 2.6 [Ref: 4.9]

FUEL	LEL (%Vol)	UEL (%Vol)
Ethane	3.5	15.1
Ethylene	2.7	34.0
Carbon Monoxide	12.5	74.0
Methane	4.6	14.2
Methanol	6.4	37.0
Pentane	1.4	7.8
Propane	2.4	7.5
Toluene	1.2	7.0
Hydrogen	4.0	76.0

Typical Explosion Limits of Flammable Gases and Vapours (Normal Temperature and Pressure) Table 2.3 [Ref: 4.8]

This range varies, but in the case of the "technical dusts" as used in research in this area typical figures are:-

LEL - 20 to 60 g/m^3
UEL - 2 to 6 kg/m^3

These limits for gases, vapours, and dusts can be affected in three main ways in modified atmospheres within hyperbaric vessels.

(1) Ignition energy can effect the explosive limits. The higher the ignition energy the wider the limits. Oxygen enriched atmospheres can create the conditions for accelerated combustion with high energy levels which initiate secondary ignition leading to an explosion.

(2) A rise in the initial pressure as in the case of a hyperbaric vessel will widen the explosive range in particular the upper limit.

(3) An increase in temperature will cause a linear enlargement of the explosive range and make it easier for the reaction to propagate.

2.4.4 Violence of Explosion

As stated earlier the violence of an explosion is indicated by the rate of pressure rise which varies according to the flammable substances involved ie. gas, vapour or dust and on the volume of the vessel. The concentration of the flammable substance must be within the explosive limits.

The relationship with the volume is characterised by the "Cubic Law" **[Ref: 4.6]** and is illustrated in **Figure 2.6 [Ref: 4.9]** for the case of coal dust with the vessel volume varied between 0.01 to 20 cubic metres.

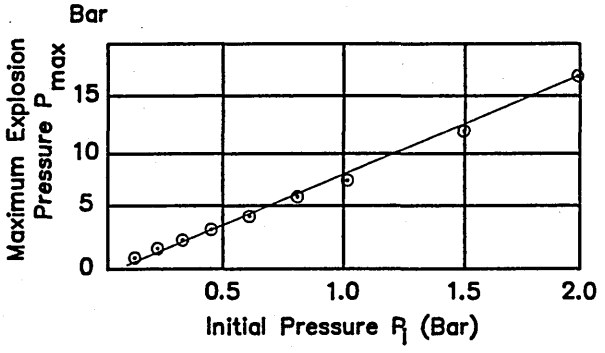
Flammable Gases $(dp/dt)_{max}\ V^{1/3} = k_g$ Eq.2.1

Combustible Dusts $(dp/dt)_{max}\ V^{1/3} = k_{st}$ Eq.2.2

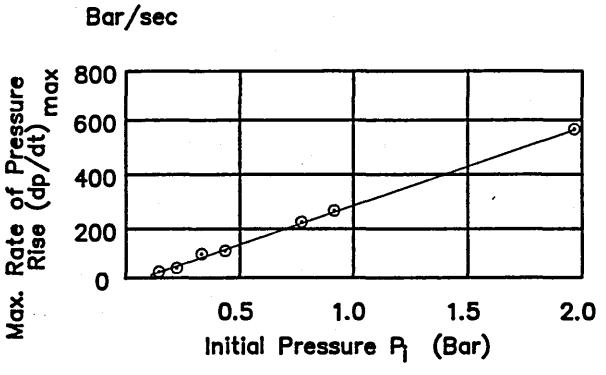
K_g is a specific material constant and for most solvents vapours falls within the range 40 to 70 bar $m.s.^{-1}$ **[Ref: 4.10]**.

K_{st} is similar to K_g except it applies to combustible dusts and typical values would fall in the range 58 to 241 bar $m.s.^{-1}$ **[Ref: 4.10]**.

If the initial pressure of the mixture within the vessel is above atmospheric pressure, as in the case of a hyperbaric vessel, then there will be a proportional increase in the maximum explosive pressure and the rate of pressure rise. **[See: Figure 2.7]**. With pressures in excess of 2 bar irregularities appear in the response with the result of a more violent explosion than expected.



Influence of Initial Pressure on Propane Explosions
FIGURE 2.7(a) [Ref: 4.11]



Influence of Initial Pressure on Propane Explosions
FIGURE 2.7(b) [Ref: 4.11]

Turbulence in the mixture has a major effect on the explosion. In the case of a gas/air mixture high levels of turbulence can dramatically increase the violence of the explosion. Increasing the rate of pressure rise in some cases 9 fold and increasing the maximum explosion pressure by 20% [Ref: 4.12].

2.5 CONCLUSIONS

Oxygen enriched atmospheres routinely exist in industry during processes and can develop inadvertently at any time when oxygen or compressed air is transported, stored or utilised.

Frequently the data available on the quality of an inert atmosphere required for combustion control in a particular process is for ambient temperature and pressure. This data is no longer valid when oxygen enriched atmospheres are created. The general effect of these modified atmospheres is to widen the combustion range; reduce the ignition energy and ignition temperature required to initiate combustion and create accelerated combustion or more destructive explosions.

In a combustion hazard analysis for an industrial application it is not only the process materials that have to be considered, but the effect of other material that may enter the process vessel. These may ignite with low ignition energy levels and provide sufficient energy for the secondary ignition of other materials.

The hazard of an oxygen enriched atmosphere can be reduced by the creation of an inert atmosphere, but with higher concentrations of inert gas or agent than suggested in Chapter 1. To determine accurately the levels required it is necessary to conduct experiments as there is little data available on the combustion

characteristics of materials subjected to oxygen enriched atmospheres in industrial processes.

Failure to understand the behaviour of materials in an oxygen enriched atmosphere and to achieve the necessary concentration of inert gas or agent can create a serious combustion hazard resulting in a fire or explosion disrupting production and endangering life.

CHAPTER 3

MIXING PROCESS AND MODEL DEVELOPMENT

3.0 INTRODUCTION

After deciding on the composition of the inert atmosphere for an industrial process the next step is to assess how much inert gas is required to create the desired atmosphere.

The current design techniques are based on a model considering the exponential rise in concentration of inert gas within a vessel or alternatively an exponential decay of the original gases as the inert gas is introduced. The controlling factors in the model are the volume of the vessel and the flow rate of inert gas into the vessel.

This model is applied in a simplistic way and it tends to be inaccurate as it does not consider additional significant factors which can have a major influence on mixing within a vessel such as gas movement, inlet position and the effect of sub-volumes.

In this chapter these factors will be considered and the effect of mean gas velocity and turbulence on the mixing within a vessel will be reviewed. A computer modelling technique will be proposed that will give the engineer, during the initial design stages, an improved insight to the likely behaviour of a system. For a detailed analysis and accurate data on the performance of a system specific models based on experimental data are suggested. These models can then be incorporated within plant simulation models to determine the total system performance.

3.1 VELOCITY AND TURBULENCE

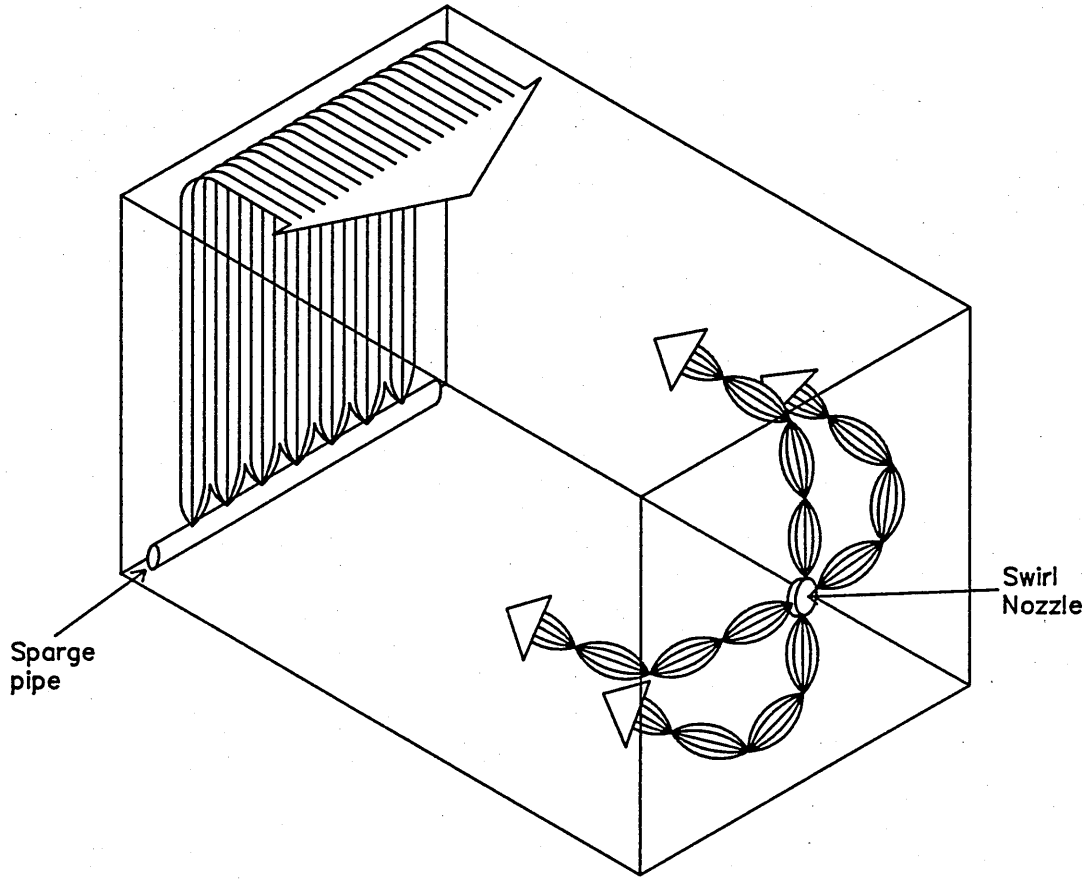
It is obvious that gas movement plays a vital role in determining the mixing rate within a vessel. This movement is assessed by measuring the velocity and turbulence intensities throughout the vessel. It is reasonable to assume that if there is a location where the velocity and turbulence is low are negligible adequate mixing will not take place in that area.

3.1.1 Inlet Type and Configuration

A common practice for introducing inert gas into a vessel is to provide a simple inlet such as a sparge pipe [See Figure 3.1]. The mixing can however be considerably improved by modifications to the inlet such as the introduction of a nozzle.

A study comparing the performance of the sparge pipe to the swirl nozzle has been conducted by Robertson [Ref: 8.0]. The conclusion after comprehensive tests was that a swirl nozzle purge system was a definite improvement over the sparge pipe, with better purging, which was more consistent and provided some insensitivity to the location of equipment within the vessel. The research indicated the reason for this improved performance was the increased turbulence created by the relatively high velocity jet streams from the swirl nozzle.

It may be the case, particularly in large vessels, that one inlet would be insufficient to create the required mixing characteristic. This is because a larger vessel is usually more complicated with divisions or large obstructions. The jet streams created by the nozzle will dissipate their energy in a portion of the volume. To overcome these problems multiple inlets are required each fitted with a nozzle and positioned in such a way to create the required movement throughout the vessel.



Relative Gas Movement from a Sparge Pipe
and a Swirl Nozzle

FIGURE 3.1

3.1.2 Fan Assisted Mixing

There are occasions when the effect of gas flowing into the vessel through the inlets will not induce sufficient gas movement and it is then necessary to assist the mixing process with a fan.

Experiments were carried out during this study to determine the effect of fan assisted mixing. The results show that with large vessel volumes or low inert gas flow rates at the inlet the mixing can be poor, but if a fan is introduced high gas velocities and turbulence are created and then effective mixing can be assured. **[See: Section 4.3]**.

This approach is adopted in some fire extinguishing applications where in large rooms the inert gas is introduced by a swirl nozzle and fans are started to improve the mixing time characteristic by increasing the mean gas velocity and creating high turbulence levels.

3.1.3 Continued Mixing

After the inert atmosphere of the desired quality has been created it is important to sustain gas movement to ensure that any leak of oxygen into the vessel is diluted and is detected by the sampling system.

The mixing can be induced by continuing the flow of inert gas into and out of the vessel. This is known as the **Continuous Flow Method** and it has the advantage of preventing any accumulation of contaminant, but inert gas is always going to waste and therefore it can be inefficient and wasteful.

There are occasions where it is necessary to seal the vessel after the inert atmosphere has been created. To

dilute contaminants and ensure that any sample taken is representative of the atmosphere within the vessel it is important to ensure adequate gas movement and mixing. This movement can be provided by a fan. This is known as the **Fixed Volume Method.** The main disadvantage is that it does not prevent the accumulation of a contaminant within the vessel, but there is the advantage that it does not waste inert gas unnecessarily.

3.2 BASIC MODEL

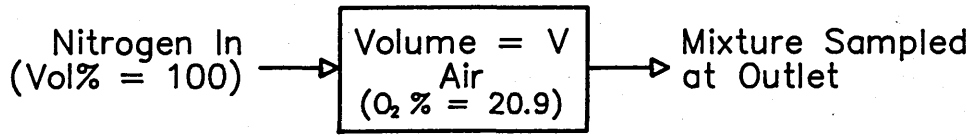
The basic model currently used in the design of inert atmospheres is based on taking the whole volume and assuming a perfect mix with an exponential rise in the concentration of inert gas within the vessel as the inert gas is introduced at the inlet. **[See: Figure 3.2].**

The equations which described the behaviour of the rise in concentration of inert gas or the decay of the original gases are given in **Figure 3.2.**

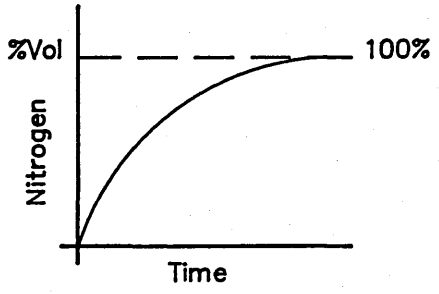
The derivation of these equations is available in a wide range of texts, typically **Reference 9.1.**

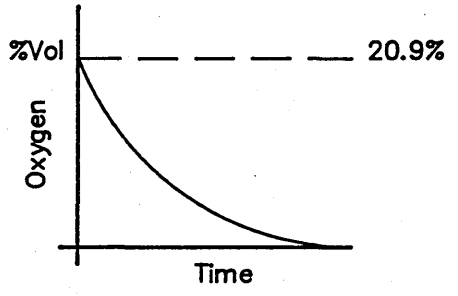
With this model it is assumed there is an instantaneous response in the entire volume as soon as the inert gas appears at the inlet. The response always takes the same general form, but the time taken to reach the final value is dictated by the time constant.

A Simple Clear Volume



At Time = 0





$C_{inert} = C_i (1 - e^{-t/T})$

[EQUATION : 3.1]

$C_{oxygen} = C_{it} e^{-t/T}$

[EQUATION : 3.2]

WHERE:–
- C_{inert} = Inert Gas Concentration at Time t
- C_{oxygen} = Oxygen Concentration at Time t
- C_i = Inert Gas Input Concentration
- C_{it} = Initial Oxygen Concentration
- t = Time (Seconds)
- T = V/Q = Time Constant
- V = Vessel Volume
- Q = Volume Flow Rate of Inert Gas

The Typical Perfect Mix Response of a Simple Clear Volume
FIGURE 3.2

3.3 VALIDITY OF THE BASIC MODEL

It is contended in this Thesis that the method by which this basic model is currently applied in industry is too simplistic and tends to the inaccurate estimation of the inert gas required to create a particular atmosphere within a vessel.

Perfect mixing as currently applied in a simple model can only be reasonably assumed when:-

1. The vessel is symmetrical[2].

If this is not the case, as in a tube, the response may approximate to "plug flow" with a long time lag followed by a fast rise to the steady state value.

2. The gas movement within the vessel must have high mean velocities and high turbulence levels.

Results from experiments during this study have shown that where this is not the case the response is erratic.

3. The vessel has one simple volume.

If there are divisions within the vessel creating sub-volumes each will have its own response. The situation becomes more complicated if there is a time lag in gas movement between sub-volumes.

4. There are no short circuits.

The gas inlet and outlets must be positioned to ensure that all the inert gas mixes with the original gases in the vessel. If these points are positioned beside each other or in the same sub-volume a significant portion of the inert gas may pass directly from the inlet to the outlet.

---

[2] Symmetrical definition = vessel width approx. equals the lenght

3.4 LUMPED PARAMETER MODELS

Although it may not be valid to use a perfect mixing model consisting of one large volume it may be possible to improve the accuracy by considering a vessel as a number of smaller unit volumes each with perfect mixing and linked in parallel or series.

This is particularly so when there are physical barriers within the vessel and it is possible to define the sub-volumes in such a way that it is valid to make the assumption of perfect mixing within each. A further assumption can then be made that there is no time lag in gas movement between the sub-volumes. If this is clearly not the case then care should be taken as large time lags will lead to system instability and erratic results. **[Ref: 10.0]**

This approach to modelling, where the system variable does not vary with position in the unit, is known as the **Lumped Parameter Model.** The definition is a model with a zero dimensional state described by algebraic equations if at steady state or by ordinary differential equations if in an unsteady state. In this application the independent variable is time and the dependent variable is the concentration of oxygen or nitrogen.

The model currently used by industry consisting of one large volume is a lumped parameter model. The recommendation made in this work is that each application should be examined carefully and the volume broken down into smaller volumes which more accurately conform to the perfect mix model. These are then linked together by gas movement between the smaller volumes.

3.5 SERIES VOLUMES

A typical example of a multiple lumped parameter model with the elements in series is a tray distilitation column [See: Figure 3.3].

Figure 3.3 illustrates the lumped parameter model and the predicted system response. The equations which predict the response are also given and these are included in a computer programme.

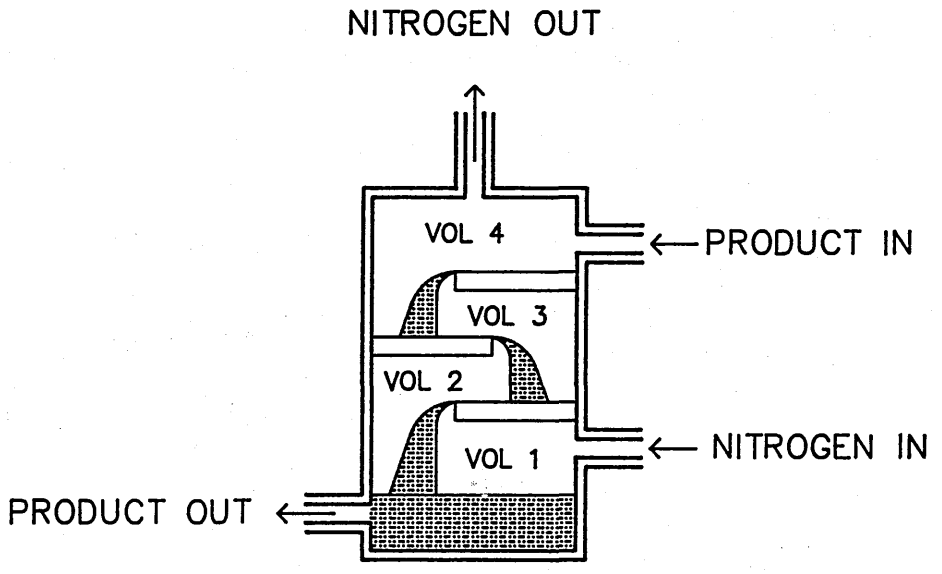
Definition of Symbols in Figure 3.3

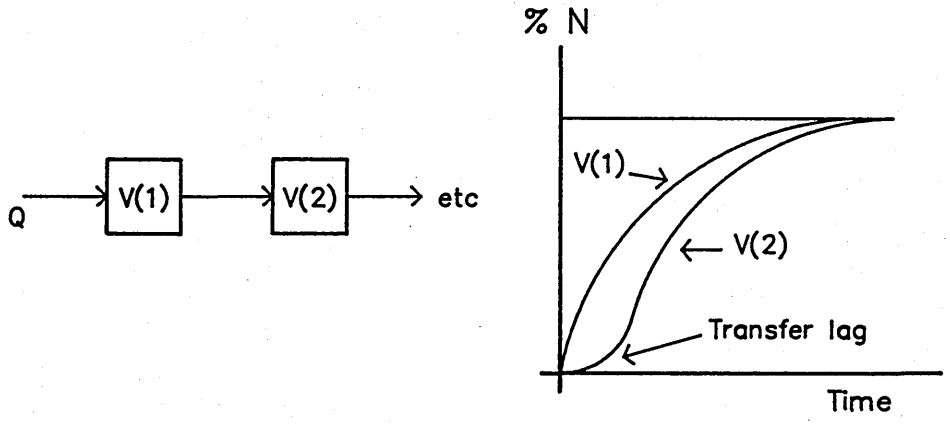
V(x) = sub-volume "x" Volume (m^3)
dt = time increment (seconds)
Q = Volume flow rate of inert gas
CNi = % Concentration of inert gas at input
C_{Nx} = % Concentration of inert gas in volume "x"
t = Time (Seconds)
t+1 = Time + one time increment (dt) (seconds)

It is assumed the tray distillation column processes material which under normal circumstances is non flammable, but in certain conditions can release a vapour. When this vapour is mixed with air it can form a flammable mixture. If sensors indicate the possibility of a hazardous condition nitrogen is injected to the system expelling the air and rendering the atmosphere within the column inert.

An engineer designing such a system would wish to determine the time taken to purge the vessel, how much gas will be required and predict the response of each sub-volume.

The system responses for two sub-volumes are illustrated in **Figure 3.3** and they display the classic form for series elements with a transfer lag developing in the response of each subsequent sub-volume. This





Volume (1) Response Equation:–

$$C_{N1}{}_{t+1} = C_{N1}{}_{t} + \frac{Q\ dt}{V(1)}\ (CNi - C_{N1}{}_{t})$$ Equation 3.3

Volume (2) Response Equation:–

$$C_{N2}{}_{t+1} = C_{N2}{}_{t} + \frac{Q\ dt}{V(2)}\ (C_{N1}{}_{t+1} - C_{N2}{}_{t})$$ Equation 3.4

Typical Response of a Series Lumped Parameter Model

FIGURE 3.3

transfer lag is an important factor and must be considered as it can play a significant role particularly when the number of sub-volumes increases. The amount of gas introduced to the first volumes may be far in excess of that required to create the necessary atmospheric conditions within this volume, but the supply must be maintained as the later sub-volumes would not reach the specified level until sometime later due to the transfer lag between the volumes. It may be more efficient to inject the nitrogen into each sub-volume but still use the same total volume of nitrogen. This would reduce the effect of the transfer lag although the response of the earlier sub-volumes would be slower.

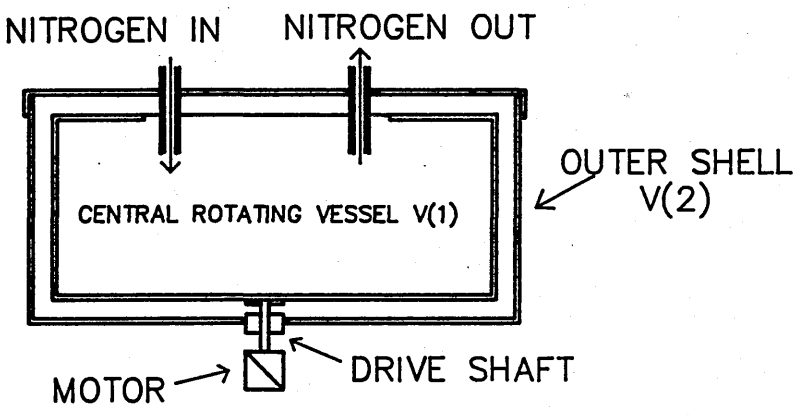
This aspect will be examined in more detail later in relation to parallel volumes. [See: Section 3.7].

3.6 PARALLEL VOLUMES

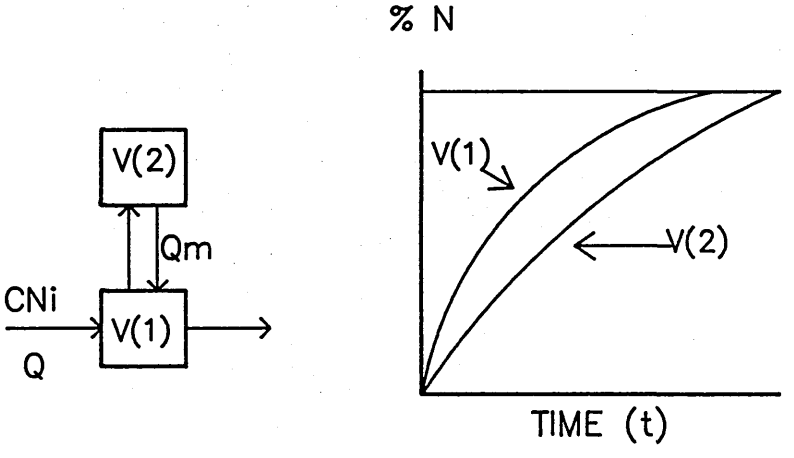
The sub-volumes may also be connected in parallel which will result in a different system response without a transfer lag. The example of a batch centrifuge is taken with the relevant aspects illustrated in Figure 3.4.

Definition of Symbols in Figure 3.4

V(x) = Sub-volume "x" volume (m^3)
dt = Time increment (Seconds)
Q = Inert gas flow rate into the system
Qm = Inert gas mixing rate between sub volumes
CNi = % Concentration of inert gas input
C_{NX} = % Concentration of inert gas in volume "x"
t = Time (Seconds)
t+1 = Time + one time increment (dt) (seconds)



BATCH CENTRIFUGE



PARALLEL VOLUME MODEL AND RESPONSE

Volume (1) Response Equation

$$C_{N1_{t+1}} = C_{N1_t} + \frac{dt}{V(1)}\left[Q(CNi - C_{N1_t}) - Q_M (C_{N1_t} - C_{N2_t})\right] \quad \text{Equation 3.5}$$

Volume (2) Response Equation

$$C_{N2_{t+1}} = C_{N2_t} + \frac{dt}{V(2)}\left[Q_M (C_{N1_t} - C_{N2_t})\right] \quad \text{Equation 3.6}$$

Typical Response of a Parallel Lumped Parameter Model

FIGURE 3.4

The centrifuge processes material which releases a flammable vapour. To prevent ignition an inert atmosphere is created within the centrifuge prior to every batch.

The lumped parameter model and response are illustrated in **Figure 3.4** with the system equations which are used within a computer programme to predict the response.

The aim in applying this lumped parameter model would be to determine how much gas is required to create the specific inert atmosphere, but also what is the effect of a leak and what is the response of each sub-volume.

Using the model an engineer can consider these points and assess the effect of alternative plant configurations. These aspects will now be examined in Section. 3.7

3.7 LUMPED PARAMETER MODEL EXAMPLE

To illustrate how useful the technique of multiple lumped parameter modelling can be during the initial design stage of an inert gas system the example of the batch centrifuge will be reviewed. **[See: Section 3.6, Figure 3.4 and Figure 3.5].**

There is a combustion hazard during this process as the materials in the centrifuge release a volatile vapour which if allowed to mix with oxygen can ignite in certain circumstances. To avoid this problem the vessel is purged with nitrogen at the start of every process to reduce the oxygen content to 9% and then the vessel is sealed.

The model suggested consists of two sub-volumes. One represents the rotating inner vessel in which the materials are processed and another which represents the space between the inner vessel and the outer shell.

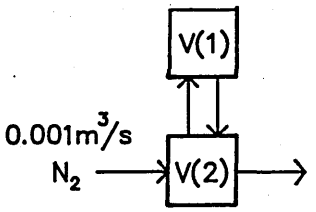
These sub-volumes are connected together in such a way that the sub volume which represents the space between the inner vessel and outer shell is connected in parallel with the rotating inner vessel. [See: Figure 3.5].

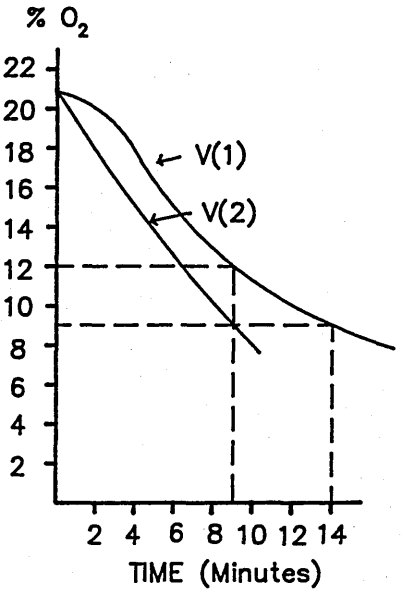
The initial design proposal is to provide one inlet introducing inert gas into the rotating inner vessel with an outlet in the same volume. The quality of the atmosphere is measured on the basis of a sample taken from the gases escaping through the outlet during the purging sequence. The nitrogen supply rate is $0.001m^3$/second and the mixing rate between the sub-volumes is assumed to be $0.001m^3$/second without any time lag in the transfer between sub-volumes. Perfect mixing is assumed in each sub-volume.

The model is processed by the computer programme and the results are as shown graphically in **Figure 3.5 (a)**. The oxygen concentration in the rotating inner vessel is reduced to 9% in 9 minutes, but at the same time the concentration in the parallel volume representing the space between the inner vessel and the outer shell is 12%. The safety level of 9% would not be reached in this outer volume until 14 minutes after the purging started, and it is possible that a dangerous mixture could accumulate in this area.

The model is now modified with two inlets, one into each sub-volume with no time lag in the transfer between sub-volumes. The inert gas supply rate is as before, but in this case the $0.001m^3$/second supply rate is divided between the two inlets in proportion to the volume of the sub-volumes. **[See: Figure 3.5 (b)]**.

The results from this model are shown in **Figure 3.5 (b)** and indicate that the oxygen concentration in the two

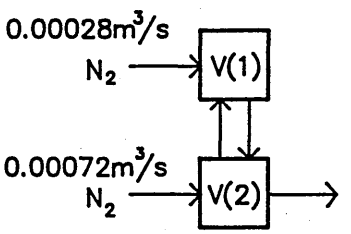


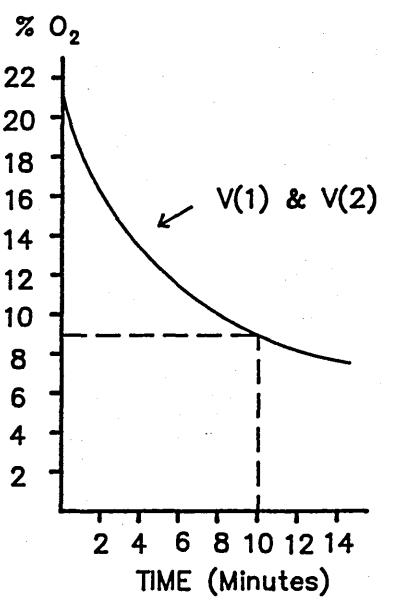


V(1)=OUTER SHELL

V(2)=INNER VESSEL

Lumped Parameter Example, Schematic and Response
FIGURE 3.5a





V(1)=OUTER SHELL

V(2)=INNER VESSEL

Lumped Parameter Example, Schematic and Response
FIGURE 3.5b

volumes reduces in unison. The safety level of 9% oxygen is reached in all areas of the centrifuge in 10 minutes. This is a slower response than the first model in relation to the inner vessel, but a great improvement when taken in relation to the complete centrifuge as in the first model it would have taken 14 minutes to reduce the oxygen concentration to 9% throughout the vessel.

The example illustrates the advantage of the lumped parameter model. It gives the engineer an opportunity to consider the likely mechanisms during the mixing process. In this case it was possible to determine the approximate quantity of inert gas required to create the desired atmosphere and indicate that the two inlet configuration is more economical in regard gas consumption than one inlet.

It is possible to extend the analysis and modify other variables to see the effect such as the mixing rate between volumes or introduce a time lag in the transfer of gas from one sub-volume to another. In the case examined here the response of the one inlet model could have been considerably improved if it were possible to increase the mixing rate between the rotating inner vessel and the space between it and the outer shell.

The accuracy of the results may be limited but they can provide indications that are a starting point for a detailed design and the results can point towards problem areas. This is clearly illustrated in this example. With the case of one inlet the state of the atmosphere is determined on the basis of a sample taken from the outlet. This outlet is connected to the inner vessel, but the results do not represent the condition of the space between the inner vessel and the outer shell. This discrepancy has thus been highlighted and remedies can then be considered.

The options are:-

1. Increase the number of sampling points.

2. Improve the mixing rate between the sub-volumes.

3. Replace one inlet with multiple inlets.

4. Incorporate safety factors in the working practice and purge for a longer period with lower oxygen safety levels.

3.8 SPECIFIC LUMPED PARAMETER MODEL

To improve the accuracy of the lumped parameter model it is possible to combine the technique with data collected from experiments carried out on real plant or scale models. The models developed in this way are specific to the particular application, but they can provide an accurate insight to the behaviour of a real system, and are of excellent value when considering the options that may improve the performance of the system.

In theory it is possible to model any system response with a combination of sub-volumes in series and parallel. The series elements create the time lag while the parallel elements determine the shape of the response. In practice this is of little use unless the sub-volume configuration reflects the actual system. If the combination of sub-volumes is created merely to conform to the system output response then the model does not necessarily predict the behaviour of the mixing process between the vessel sub-volumes.

This method of modelling is only accurate when clearly identified physical divisions exist within a vessel and careful experiments conducted to measure the gas

movement within the vessel. If these conditions are met the method provides an accurate means of determining, for a specific case, the optimum inlet configuration for the inert gas, how much gas movement is required and the response of the system should there be a leak of contaminant into the vessel.

In Chapter 4 this approach will be adopted during experimental work on a model autoclave to determine the accuracy and validity of the technique.

3.9 REGRESSION ANALYSIS

This is an approach to model development as opposed to engineering design that does not require an understanding of the mixing processes within the vessel. The approach requires experimental measurement and the analysis of the results by statistical techniques.

In this case we assume the system is a "black box" and the equation relating the output to the input is derived by linear regression methods.

In general the response of a system is an exponential rise in the inert gas concentration or an exponential decay of the original gases.

When the analysis is complete the transfer function relating the independent variable to the dependent variable takes the form:-

$$Y = e^{at+b} \quad - \quad \text{Equation 3.7}$$

Where:- Y = Dependent Variable (Either oxygen or nitrogen concentration)
t = Independent variable (Time)
a = Regression Coefficient
b = Regression Constant

3.10 SUMMARY

One of the major influences on the mixing process is the mean gas velocity and turbulence levels within the vessel. To ensure effective and efficient mixing it is important to create high velocity and turbulence levels. This can be achieved by controlling the method by which the inert gas is introduced to a vessel as it can be designed to induce the necessary movement or if this is not possible a fan can assist the mixing process by improving the turbulence levels.

The current design method for inert gas systems assumes a simple clear volume model with perfect mixing, but this may not be a valid assumption in practical applications.

A better approach giving more information and improved insight into a system is available using lumped parameter models and splitting large volumes into discrete sub-volumes operating in series or parallel. The accuracy can be improved by linking the models to experimental data.

The alternative approach is to treat the system as a "black box" and determine the relationship between the input and output by linear regression. This method is

accurate, but limited information is produced and it does require the support of detailed and accurate experimental data.

The various aspects raised in this chapter will be tested in Chapter 4 with a series of experiments on two pieces of apparatus to test the validity of this theory.

CHAPTER 4

VALIDATION OF MODELS

4.0 INTRODUCTION

In Chapter 3 some of the factors involved in the mixing process were discussed and various models were suggested that describe the response of the system to a change in input.

This chapter will assess the performance of these models in a series of experiments on two pieces of apparatus. One a simple clear volume and the other a model of an autoclave.

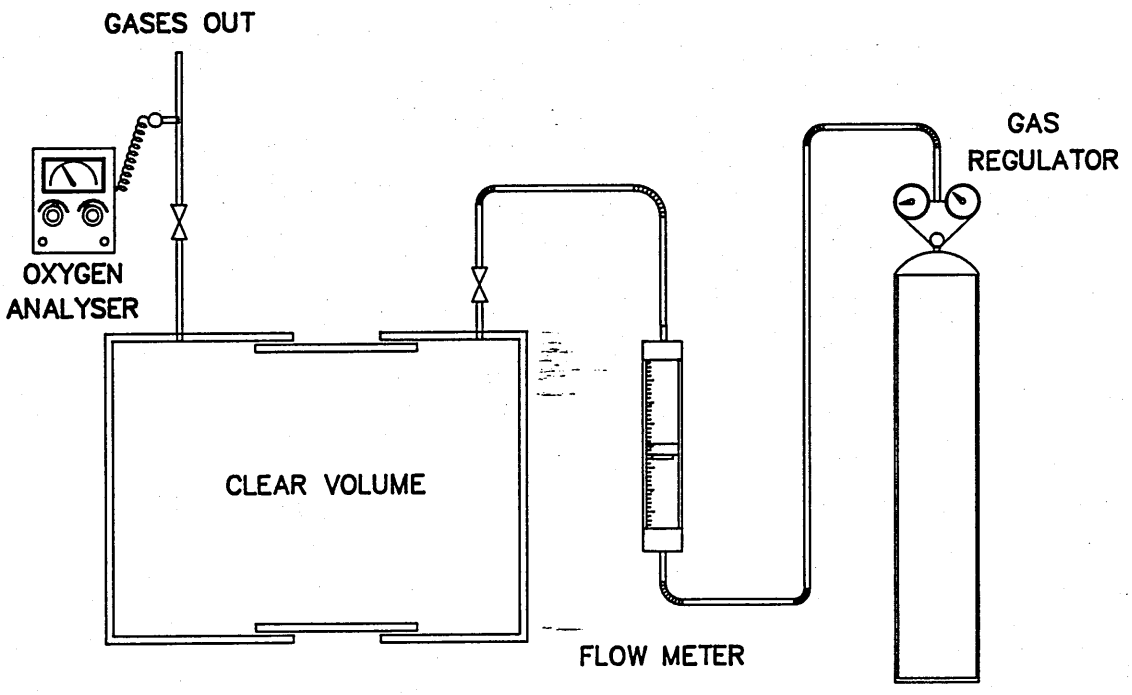
4.1 A SIMPLE CLEAR VOLUME

The perfect mix model is normally applied in a simplistic way with the response of the system assessed in relation to the input rate of inert gas and the total volume of the vessel to be purged. A series of experiments were conducted to determine the validity and accuracy of this approach.

4.1.1 Apparatus

The apparatus consisted of a cylinder which could be varied in length providing a volume between 0.292 and 0.383 m^3. **[See : Figure 4.1]**

Nitrogen gas is supplied from a high pressure cylinder with the volume flow rate controlled by a regulating valve. The nitrogen entered the vessel via a plain open ended pipe.



EXPERIMENTAL APPARATUS–SIMPLE CLEAR VOLUME
FIGURE 4.1

The oxygen concentration within the vessel atmosphere was determined by sampling the gases at the outlet tested by a Neotronics Oxygen Analyser OTOX 91.

4.1.2 Method

The length of the cylinder was set, the joints sealed with tape and the vessel initially filled with air.

The nitrogen gas was introduced at a constant volume flow rate with gas samples taken from the outlet and processed by the oxygen analyser at one minute intervals.

To increase accuracy and reduce the effect of errors the nitrogen gas flow rates were selected at random with the tests repeated at various volumes between 0.292 and 0.383 m^3.

4.1.3 Accuracy

The accuracy of the flow meter which measured the nitrogen gas flow into the vessel is + or - 10% at full scale.

There was a time lag in processing the gas samples at the outlet. This consisted of the time to transfer the gas sample through the pipework to the transducer, the time to process the sample and then provide a reading. The time lag was assessed to be 6 seconds.

It is assumed that leaks from the system were negligible. Weak points such as the sliding joints

on the cylinder were sealed prior to each experiment.

4.1.4 Results

Considerable data was collected during these experiments with the results for a volume of 383 litres taken as a representative sample for illustration here.

Appendix A has a selection of the results in a tabular form with some of the results for the 383 litre volume illustrated in **Figures 4.2** and **4.3** in this Chapter.

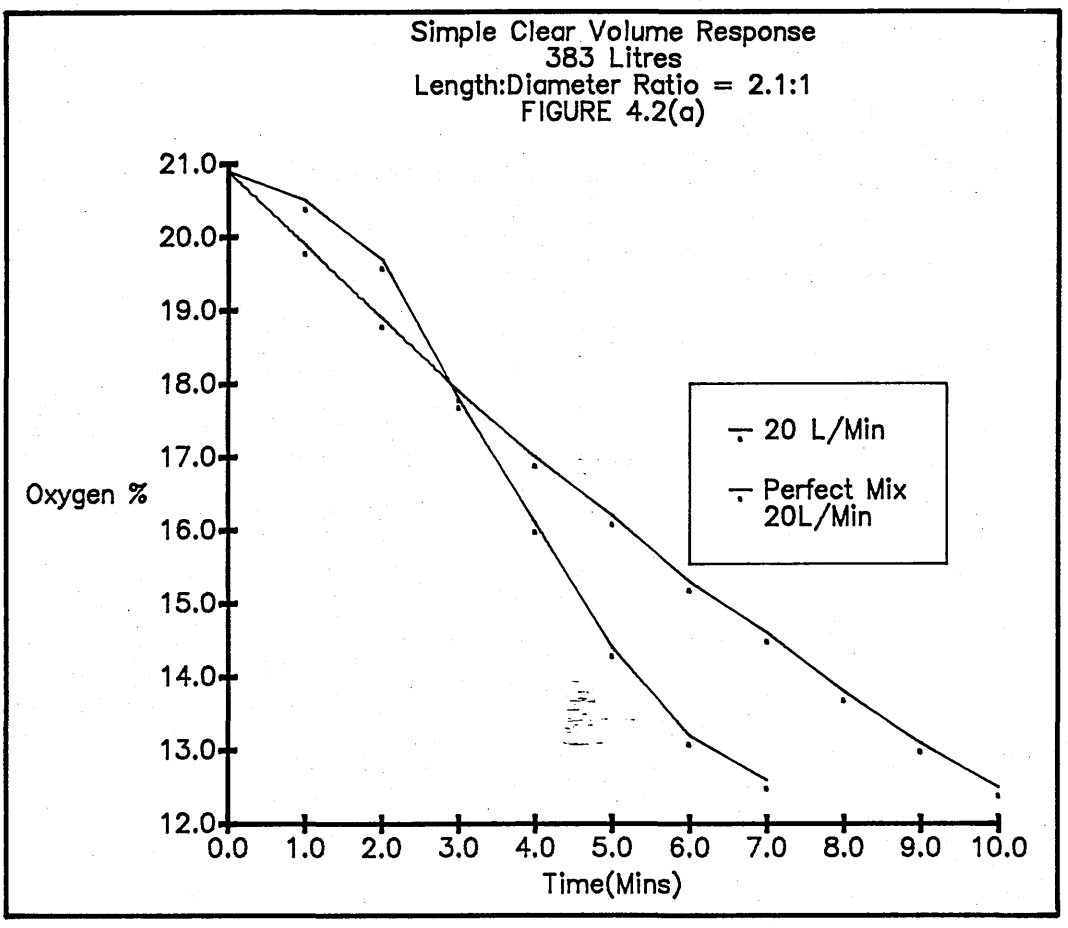
4.1.5 Discussion

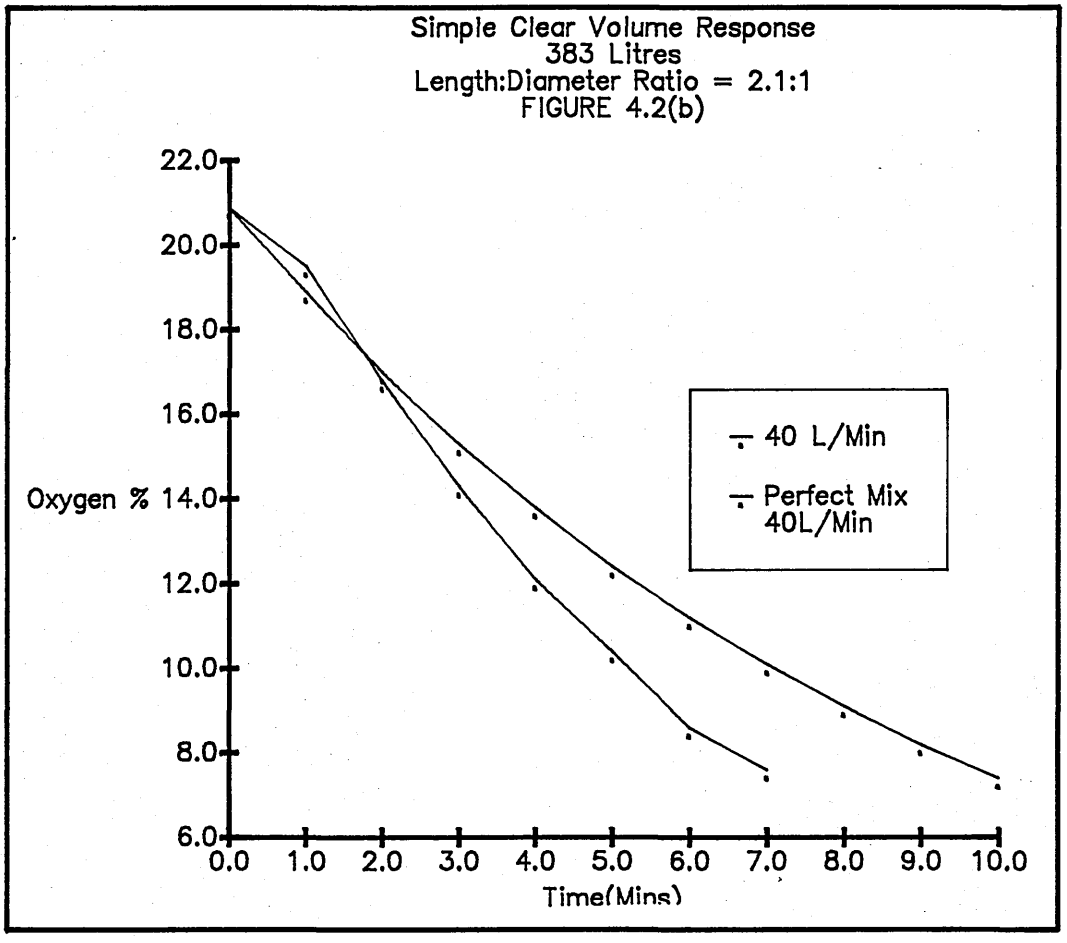
Prior to the experiments it was taken that perfect mixing could be reasonably assumed as the case complied with most of the conditions that were set in Chapter 3, **Section 3.3.**

1. The vessel is symmetrical
2. The vessel has one simple volume
3. There are no obvious short circuits for Gas movement.

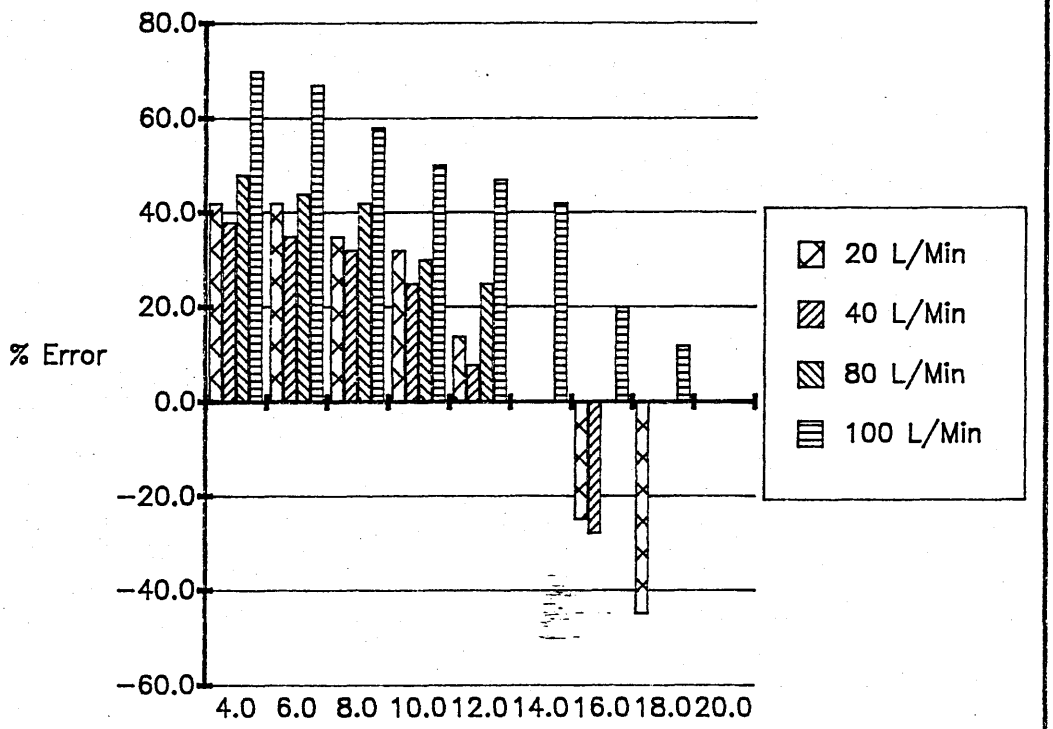
The gas movement within the vessel is dependant on the volume flow rate of inert gas into the vessel.

An examination of the results has shown that even in this apparently near ideal case the perfect mix model provides a poor approximation with a general tendency to over estimate the oxygen concentration compared to the actual value. It is assumed in this

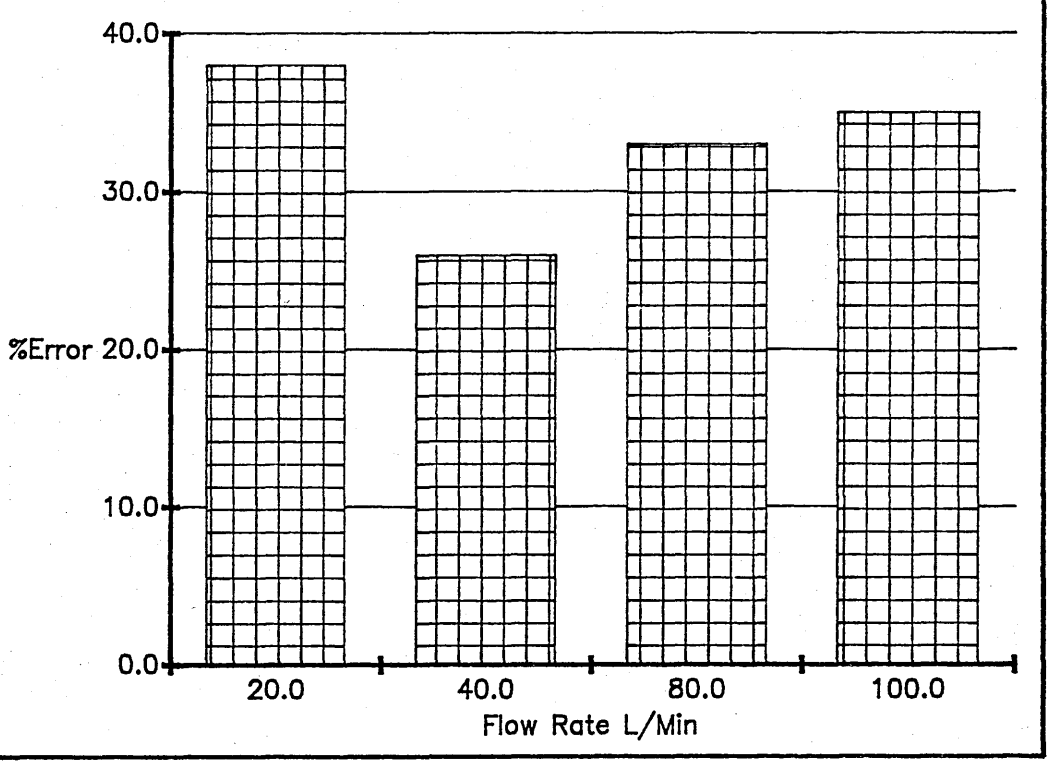

Simple Clear Volume Response
383 Litres
Length:Diameter Ratio = 2.1:1
FIGURE 4.2(a)
21.0
20.0
19.0
18.0
17.0
16.0
15.0
14.0
13.0
12.0
Oxygen %
0.0 1.0 2.0 3.0 4.0 5.0 6.0 7.0 8.0 9.0 10.0
Time(Mins)
20 L/Min
Perfect Mix
20L/Min


Simple Clear Volume Response
383 Litres
Length:Diameter Ratio = 2.1:1
FIGURE 4.2(b)
22.0
20.0
18.0
16.0
14.0
12.0
10.0
8.0
6.0
Oxygen %
0.0 1.0 2.0 3.0 4.0 5.0 6.0 7.0 8.0 9.0 10.0
Time(Mins)
40 L/Min
Perfect Mix
40L/Min

% Error in Purge Time
Perfect Mix Compared to Actual Response
FIGURE 4.3(a)



% Error in the Calculated Inert Gas Consumption
to Achieve 13% Oxygen Concentration
Consumption Based on a Perfect Mix in a Simple
Volume
FIGURE 4.3(b)



case that the actual value measured is representative of the whole volume.

Take the case of a nitrogen flow rate of 20 litres/min [See: **Figure 4.2(a)**] The perfect mix model has a faster response in the decay in oxygen concentration for the first two minutes, but after this the response is much slower with the error increasing to 44% in the estimated time to reach a 13% oxygen concentration.

The engineer would normally apply the perfect mix model in this general way at the initial stage of a design in order to determine the amount of gas required and the time to purge a system, but the errors can be considerable.

If the aim was to reduce the oxygen concentration in this apparatus to 13% by purging the system with nitrogen at 40 litres/min then the perfect mix model would over estimate the consumption by 27%. Not serious at this scale, but if applied to a real plant with repetitive purging the over estimation can have a major effect on the size and capital cost of the inert gas plant.

Figures 4.3(a) and **4.3(b)** illustrate the errors that have appeared during the analysis of the results from the experiments.

4.2 THE MODEL AUTOCLAVE

The situations that arise within industry are usually more complicated than simple clear volumes and an example will be considered here. This is a model autoclave which has sub-volumes within the vessel created by physical barriers. The aim of

these experiments is to determine the accuracy of the proposals made in Chapter 3.

The original aim was to construct a small scale model of the real autoclave, but the information available at the time of construction was inaccurate. This came to light through the appearance of experimental differences in the behaviour of the real system compared to the small model. During the subsequent close examination of the real autoclave constructional differences became apparent.

The mathematical model for the small model autoclave will therefore not be applied to the real autoclave, but rather it will be used to test the validity of applying the techniques to a real system.

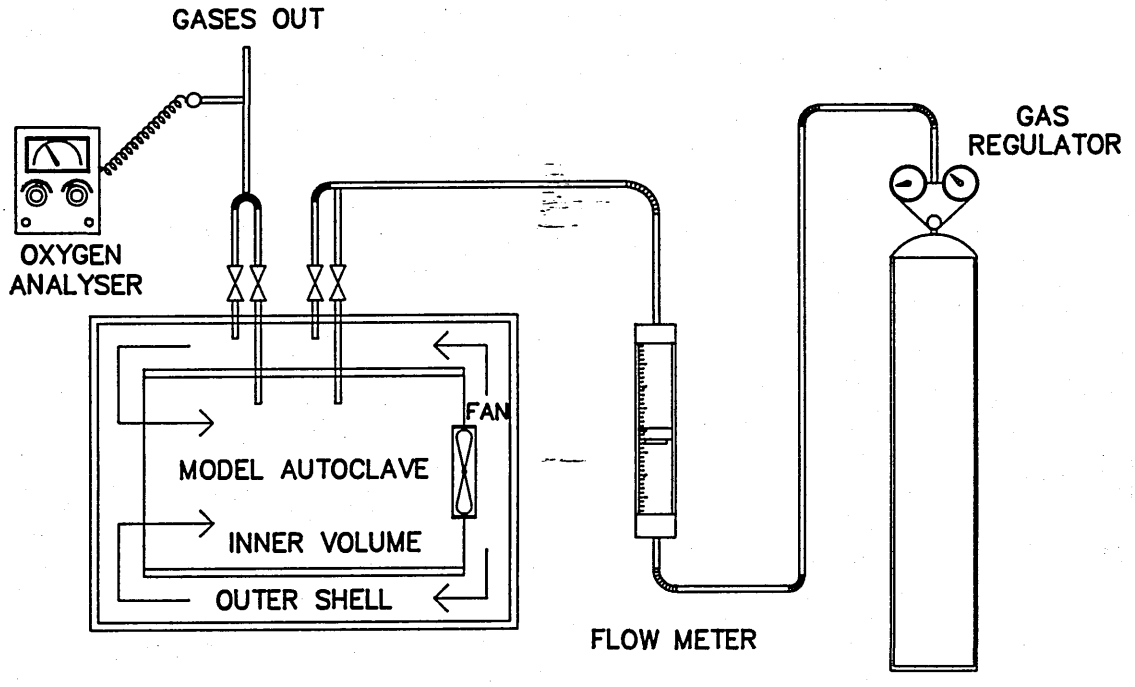
4.2.1 Apparatus

The apparatus consists of a cylinder with a volume of 0.243 m^3 metres closed at both ends and an inner cylinder open at one end and a fan at the other. **[See: Figure 4.4]**

Nitrogen gas is supplied from a high pressure cylinder with the volume flow rate controlled by a regulating valve.

The pipework is arranged so that nitrogen can be introduced to the inner volume or the outer volume with the gases escaping from either volume

The oxygen concentration in the vessel can be measured at the outlet or samples can be taken from the inner volume. **[See: Figure 4.4].**



EXPERIMENTAL APPARATUS – MODEL AUTOCLAVE
FIGURE 4.4

A fan provides the mechanical means for recirculating the gases.

4.2.2 Method

The vessel is sealed with air at atmospheric pressure and the fan is switched either ON or OFF depending on the experiment.

The experiments were conducted on a random basis and replicated to increase the accuracy and reduce the effect of errors.

In each group the nitrogen input was varied to set values in the range 20-100 litres/min. The experiments were divided into five groups (a to e) as given in sub-section 4.2.4.

4.2.3 Accuracy

The flow meter has an accuracy of + or - 10% at full scale. There is a time lag in sampling the atmosphere which was found to be 6 seconds for the outer volume and 10 seconds for samples taken from the centre of the vessel.

4.2.4 Results

Data was collected during the experiments and will now be reviewed in separate sections under the following headings:-

(a) The Effect of a Fan on Mixing
(b) The Validity of the Perfect Mix
(c) A Three Element Series Model
(d) A Two Element Parallel Model
(e) The Regression Model

Appendix B contains typical results from the experiments carried out on the model autoclave.

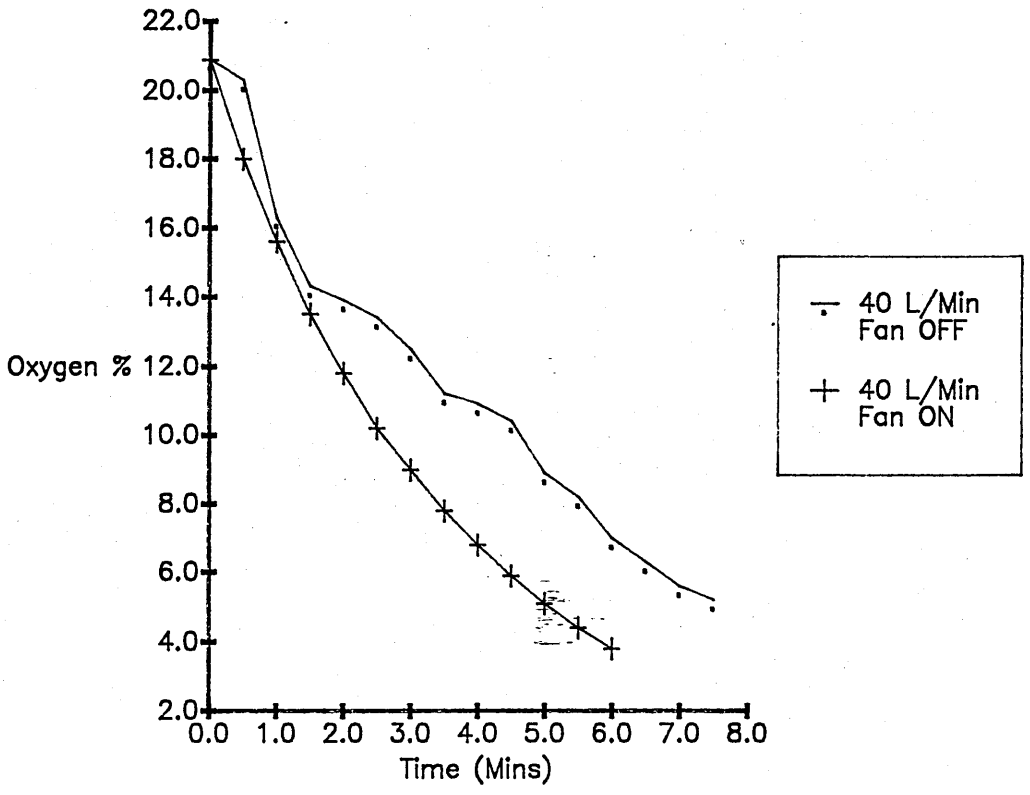
4.3 THE EFFECT OF A FAN ON MIXING

In Chapter 3 [**See: Section 3.1**] the importance of gas movement in the mixing process was identified. There are practical problems in achieving the movement in all cases particularly in large vessels or where divisions exist within vessels.

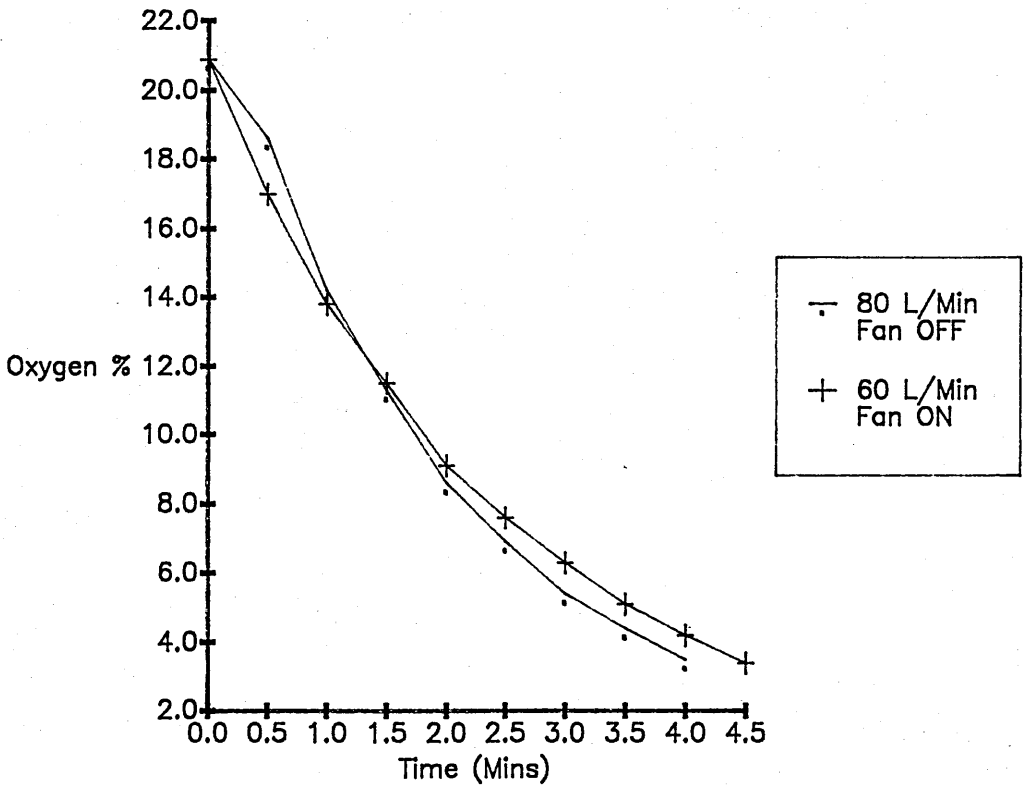
A fan can be introduced which in addition to improving the mixing within sub-volumes can ensure rapid transfer of gas between the sub-volumes. In the real autoclave [**See: Chapter 5**] the fan already exists as it is used to recirculate the gases through a heater battery to raise the temperature in the vessel.

A series of experiments were conducted with the model autoclave to determine the effect of the fan on the response of the system. Some of the results are illustrated in **Figures 4.5(a)** and **4.5(b)**.

The Effect of a Fan on the Purging Response of a Model Autoclave
FIGURE 4.5(a)



The Effect of a Fan on the Purging Response of a Model Autoclave
FIGURE 4.5(b)



As stated in **sub-section 3.1.2** the effect of a fan can be considerable, particularly with low inert gas flow rates. The improved mixing making the oxygen decay smoother and faster. This is clearly demonstrated in the experiments where it was found that there was little difference in the responses at 80 and 100 litres/minute with the fan OFF or ON. The nitrogen flowing into the vessel apparently induced effective mixing. The fan had a marked effect at 20 and 40 litres/minute.

Figure 4.5(a) shows the oxygen decay with a nitrogen flow rate of 40 litres/min. with the fan OFF and ON. For the first two minutes the responses are similar but after this they diverge. If the aim is to reduce the oxygen concentration to 6% based on a sample taken from the outlet the system with the fan running would achieve this in 4.5 minutes whereas with the fan OFF it takes 6.75 minutes.

The fan helps to create a homogeneous mixture throughout the vessel. Without the fan there can be a build-up of inert gas in the sub-volume where the nitrogen is introduced in excess of the required level, but this may not transfer to the sampled sub-volume. The system will keep the nitrogen flowing into the vessel until the sampled volume reaches the required level. This leads to inefficiency.

It was discovered that [**See: Figure 4.5(b)**] the same rate of decay in oxygen produced by a nitrogen flow rate of 80 litres/minute with the fan OFF could be achieved by a flow of only 60 litres/minute with the fan ON. This represents a considerable saving in gas consumption when the scale of the system increases and regular purges are required.

4.4 THE VALIDITY OF THE PERFECT MIX

The design of the inert gas plant for the real autoclave which will be reviewed in Chapter 5 and 6 was based on a simple perfect mix model. The interior of the vessel was taken as one volume and the flow rate was set . The amount of nitrogen gas required to reduce the oxygen concentration to certain values was then calculated on the basis of the response.

In order to assess the likely accuracy of this approach a series of experiments were conducted with the model autoclave at nitrogen flow rates of 20, 40 and 60 litres/min. The recirculation fan was ON during these tests.

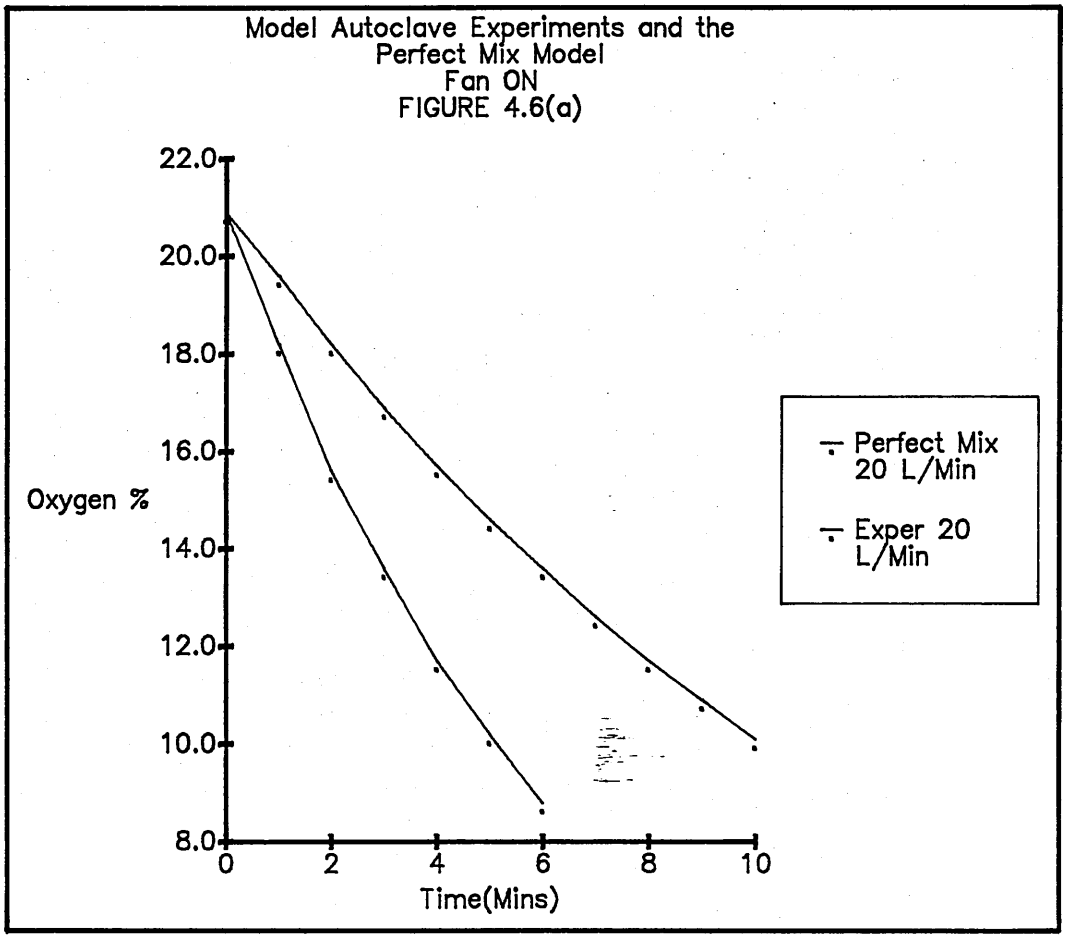
The results from these experiments were compared to the response suggested by the simple perfect mix model as applied in the design of the inert gas plant for the real autoclave.

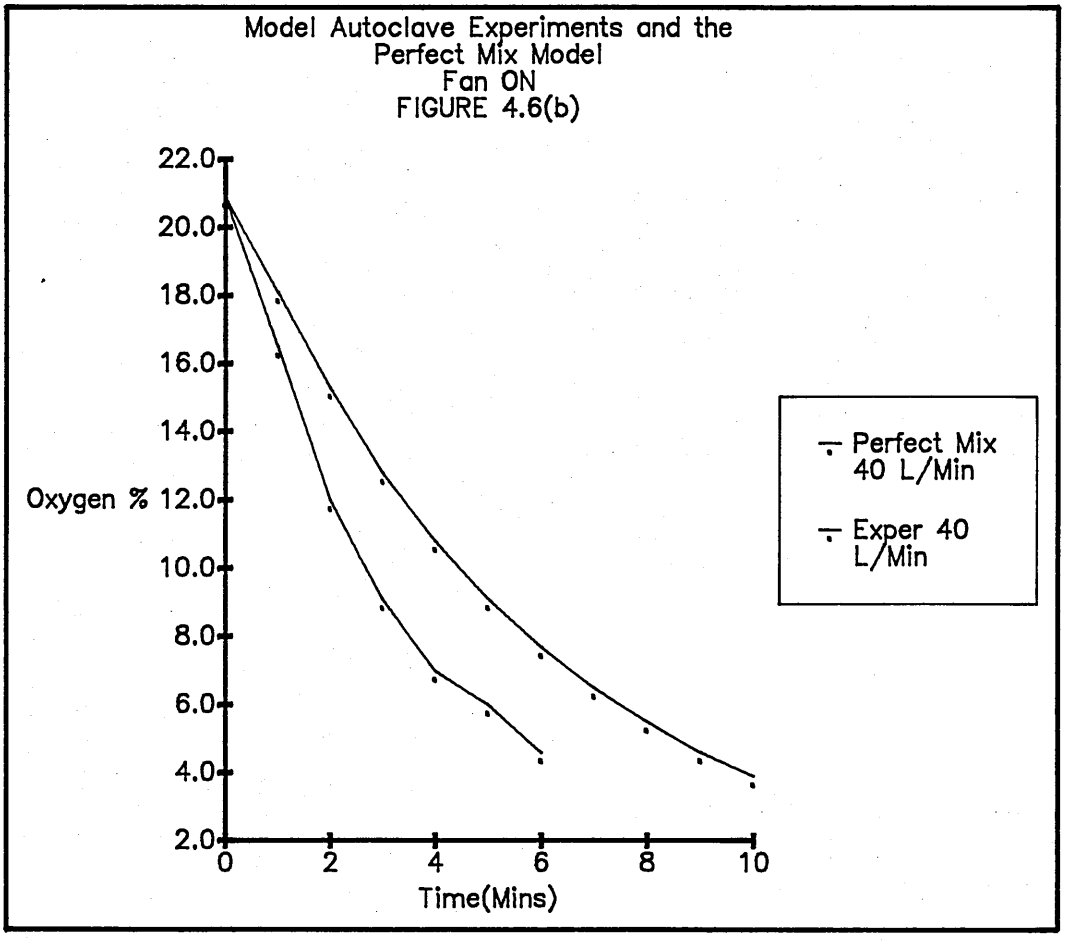
The results are illustrated in Figures 4.6(a),(b), (c) and show there is a considerable discrepancy in the two responses.

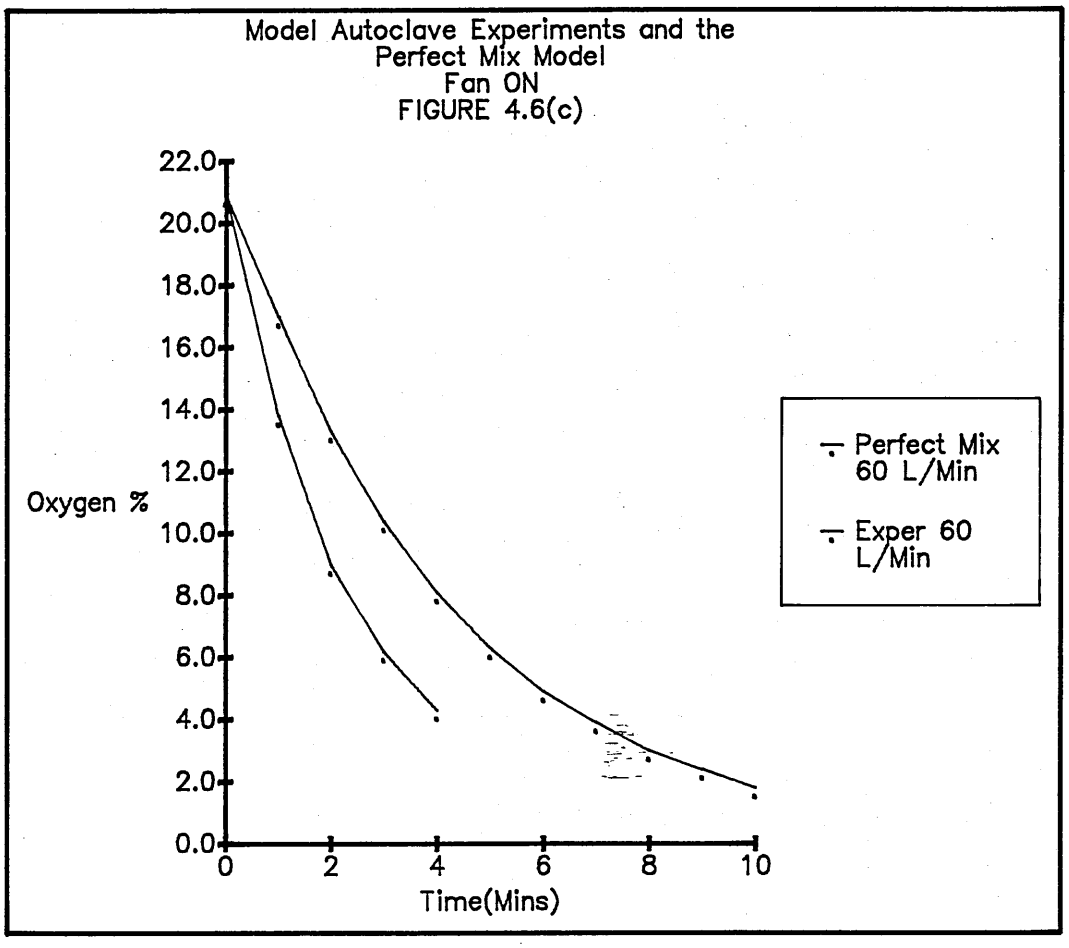
There are a number of reasons for this discrepancy principally:-

(1) The perfect mix model suggests a long mixing time due to the high time constant for the system as it has a relatively high volume compared to the nitrogen supply rate.

(2) The perfect mix model did not allow for the sub-volumes and the effect of the fan which creates effective mixing within the volumes and between the sub-volumes.


Model Autoclave Experiments and the
Perfect Mix Model
Fan ON
FIGURE 4.6(a)
22.0
20.0
18.0
16.0
14.0
12.0
10.0
8.0
Oxygen %
0
2
4
6
8
10
Time(Mins)
Perfect Mix
20 L/Min
Exper 20
L/Min


Model Autoclave Experiments and the
Perfect Mix Model
Fan ON
FIGURE 4.6(b)
22.0
20.0
18.0
16.0
14.0
12.0
10.0
8.0
6.0
4.0
2.0
Oxygen %
0
2
4
6
8
10
Time(Mins)
Perfect Mix
40 L/Min
Exper 40
L/Min


Model Autoclave Experiments and the
Perfect Mix Model
Fan ON
FIGURE 4.6(c)
Oxygen %
22.0
20.0
18.0
16.0
14.0
12.0
10.0
8.0
6.0
4.0
2.0
0.0
0
2
4
6
8
10
Time(Mins)
Perfect Mix
60 L/Min
Exper 60
L/Min

(3) The perfect mix model assumes the sample at the outlet is representative of the complete volume, but his is not the case.

(4) In the real example reviewed in Chapters 5 and 6 it is the inner volume that is of significant interest as it is here that combustion is likely to take place.

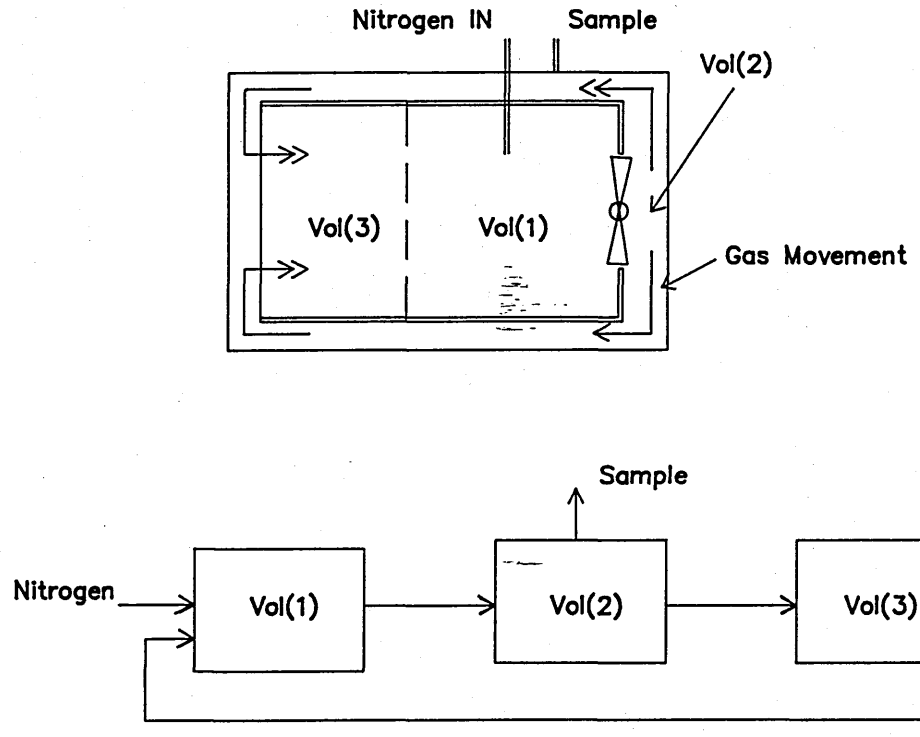
4.5 THE THREE ELEMENT SERIES MODEL

It has been suggested in Chapter 3 **[See: Section 3.4]** that the lumped parameter model approach can be improved by examining the particular case in more detail. If the structure of the vessel is considered and the probable gas movement assessed it can be possible to break the vessel down into discrete volumes linked together by gas transfer across the boundaries.

This approach was adopted in the case of the model autoclave and the experiments were carried out to test the validity of the theory.

Figure 4.7 illustrates the proposed lumped parameter model which consists of three elements in series with the nitrogen introduced to the inner volume at 40 litres/min and the vessel atmosphere sampled at the outlet from the outer volume.

The inner volume is assumed to consist of two sub-volumes. One adjacent the fan where the nitrogen is introduced and the other at the opposite end of the vessel. The nitrogen is introduced to sub-volume(1) where it mixes with the original gases and then the mixture is drawn into sub-volume (2) by the fan.



Model Autoclave 3 Series Elements
FIGURE 4.7

Sub-volume(2) is considered to represent the outer volume of the autoclave and it is from here that the outlet provides the sample for the oxygen analyser. The gases are then passed to sub-volume (3) and in turn to sub-volume (1) with the recirculation process continuing.

The results from the experiments related to the predicted response provided by the lumped parameter model are illustrated in **Figures 4.8(a),(b).**

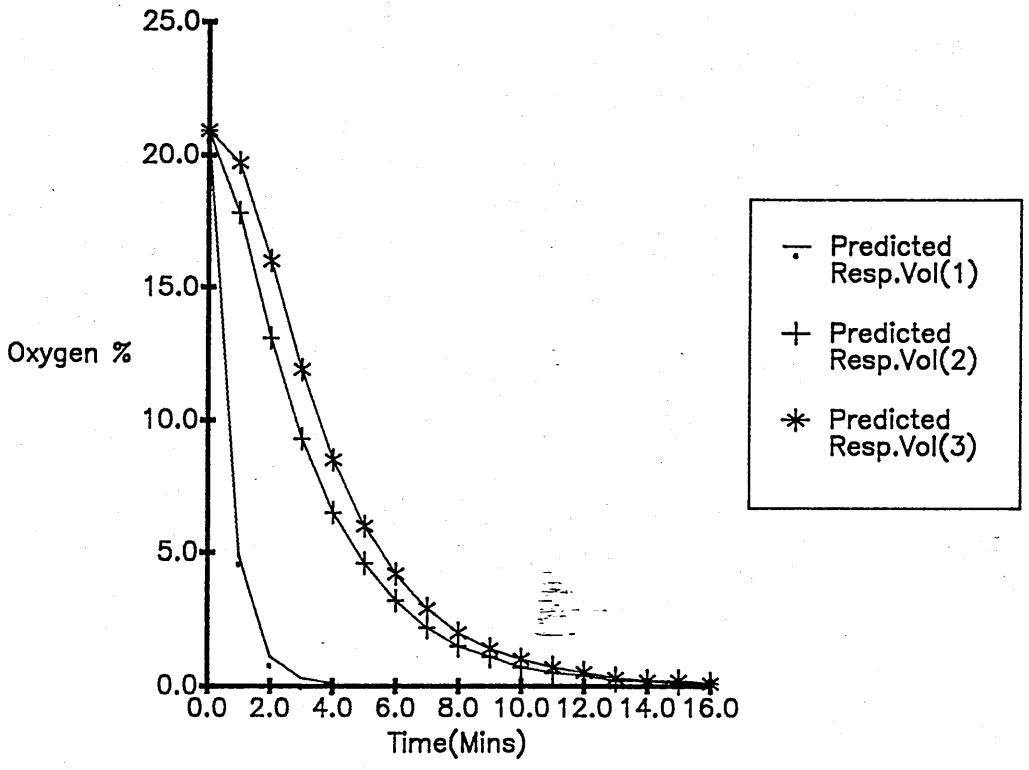
Figure 4.8(a) illustrates the predicted response in the three sub-volumes and as they are in series there is as expected an increasing time lag in the responses. The system considers the sample taken from sub-volume (2) as representative of the vessel atmosphere, but from the responses it is obvious the sub-volume(3) lags the sub-volume (2) response. This would cause some concern and further investigations with the model autoclave have indicated that the three element model is in error in two ways.

Firstly, the third sub-volume is smaller than anticipated and the response is closer to that of sub-volume (2) than suggested in **Figure 4.8(a).**

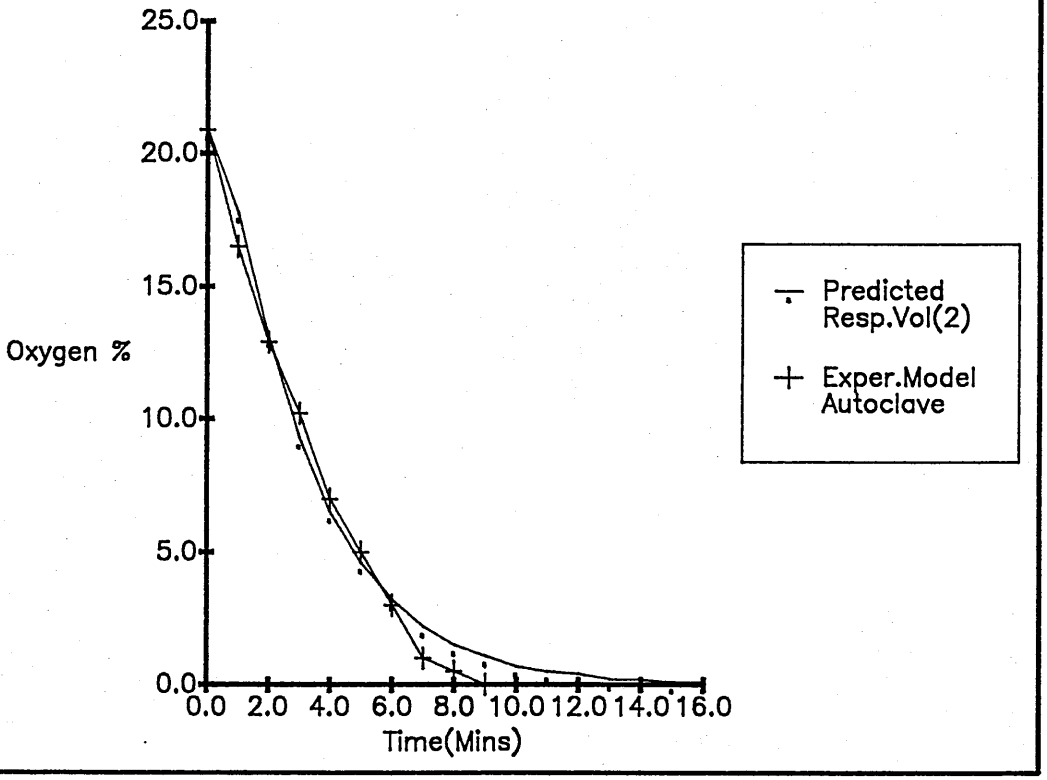
Secondly, the response of sub-volume (2) has a time lag that does not appear in the experimental data. This suggests that a model with parallel sub-volumes may provide a closer response to the actual system.

Figure 4.8(b) illustrates the predicted response at the sampling point compared to the actual response measured during the experiments. The two responses have a close similarity and represent a reasonable degree of accuracy certainly an improvement on the perfect mix model particularly at the later stages in the purge cycle.

Predicted Response
of
The 3 Element Series Computer Model
FIGURE 4.8(a)



Predicted Response of Vol(2) Compared to
Actual Response of The Model Autoclave
(3 Series Element Model)
FIGURE 4.8(b)



These experiments have shown that by breaking down a large volume into a number of sub-volumes can lead to improved accuracy and give an insight to the gas movement within a large vessel. In this case it is possible to accurately determine the amount of gas required. In addition there is the suggestion that there could have been a problem with mixing in sub-volume(3). Further investigations have shown this is not the case with the model autoclave although it is a point that should be considered when the application is scaled up to a full size autoclave.

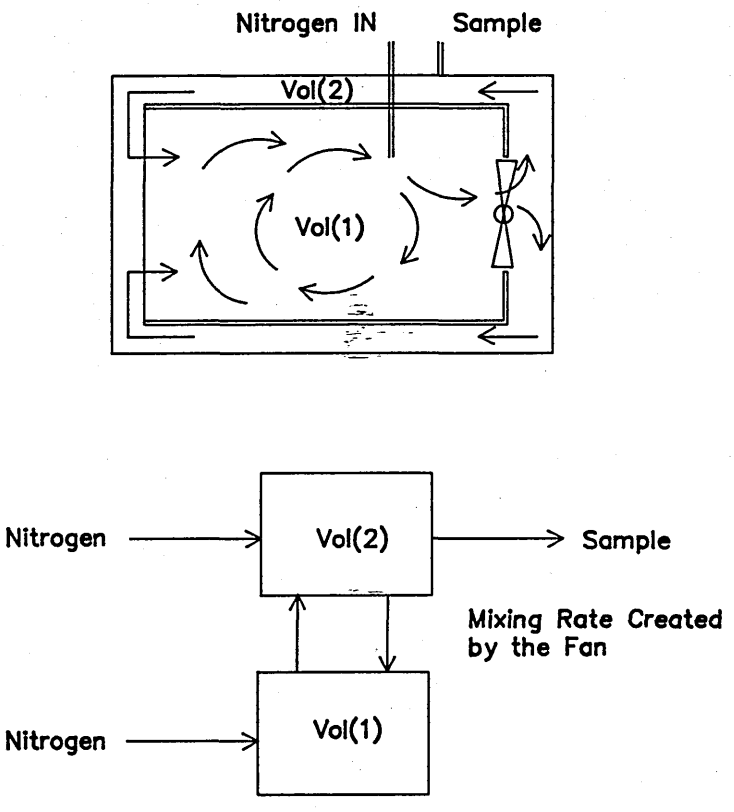
4.6 A TWO ELEMENT PARALLEL MODEL

Another configuration for the model suggested in Chapter 3 [See: Section 3.8] is one with two volumes connected in parallel.

Experiments on the model autoclave were carried out to test this theory and to determine its accuracy in predicting the response of the model.

The autoclave was set up with the inert gas introduced to the centre of the vessel, the atmosphere sampled at the outlet from the outer volume and the fan ON. Nitrogen was introduced at four flow rates 20, 40, 60, and 100 litres/min with typical results illustrated in **Figures 4.10(a),(b).**

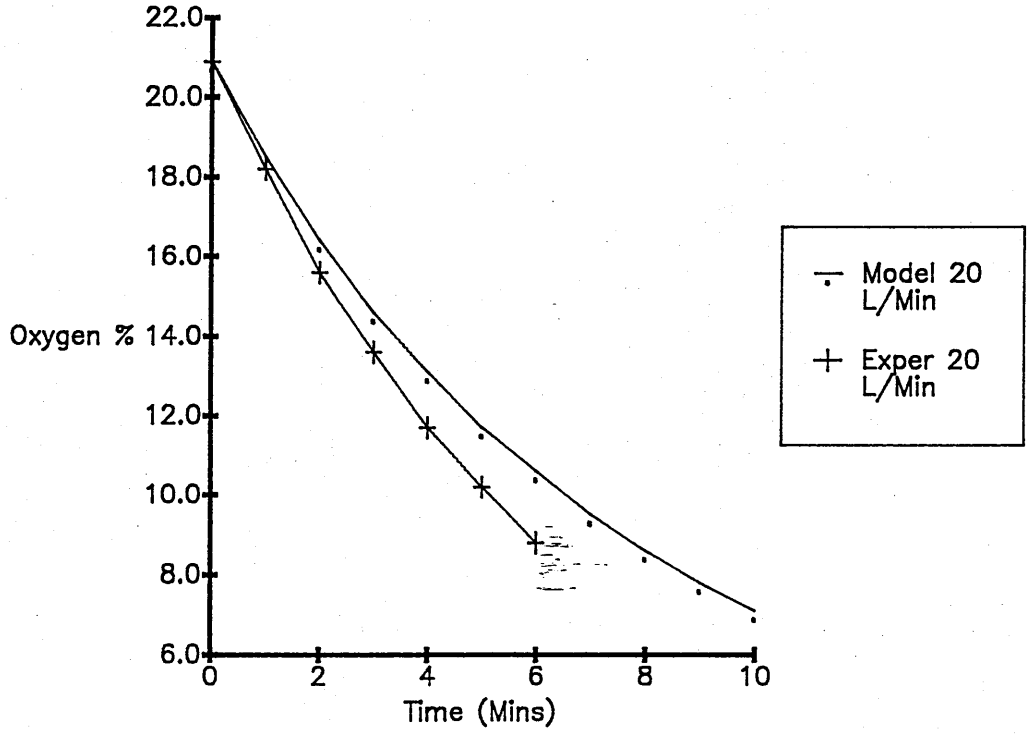
On the basis of the information gathered during these experiments the lumped parameter model proposed consists of two sub-volumes linked in parallel with a nitrogen supply into each. Sub-volume (1) represents the inner volume of the autoclave and Sub-volume (2) represents the outer volume.**[See:Figure4.9].**



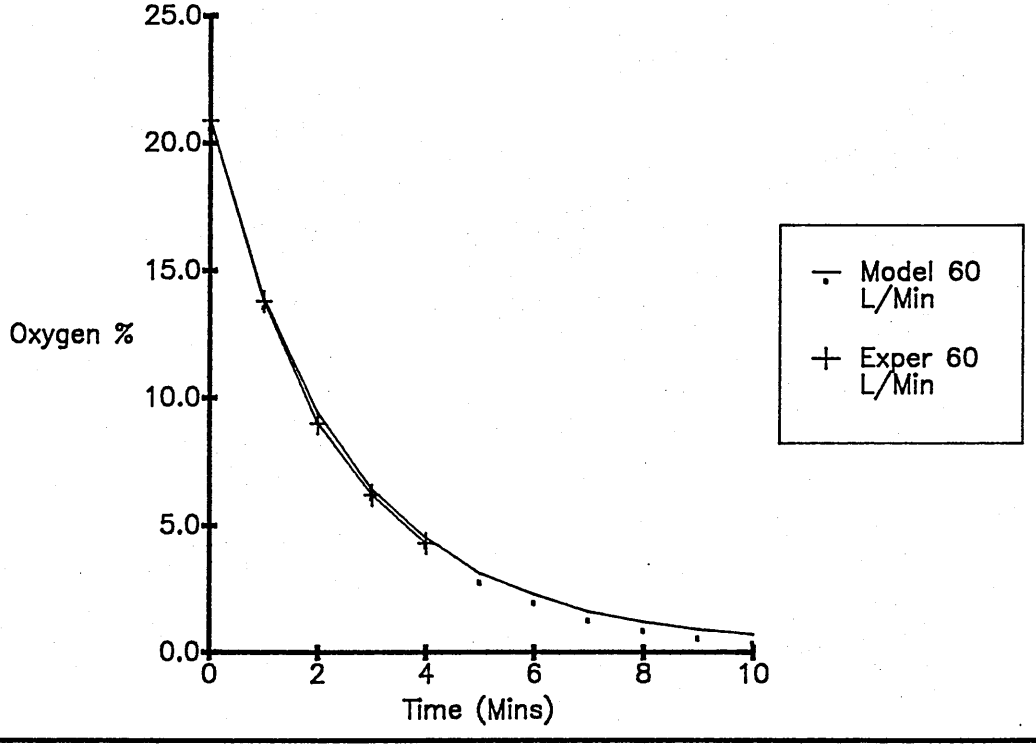
Model Autoclave 2 Parallel Elements

FIGURE 4.9

Predicted Response of Vol(2) Compared to the
Actual Response
of The Model Autoclave
(2 Parallel Element Model)
FIGURE 4.10(a)



Predicted Response of Vol(2) Compared to the
Actual Response
of The Model Autoclave
(2 Parallel Element Model)
FIGURE 4.10(b)



There is an inconsistency between the autoclave and the model suggested as there is only one nitrogen supply which is into the inner volume whilst the computer model proposed has a nitrogen inlet to both inner and outer volumes.

As the nitrogen gas inlet to the inner volume is near the fan it is suggested that a significant portion of the nitrogen entering this volume does not get time to mix with the original gases, but is transferred by the fan to sub-volume (2). The rest of the nitrogen entering the inner volume mixes with the original gases on the basis of a perfect mix and is then transferred to sub-volume (2)

The parallel model was developed by taking the results from the experiments with a nitrogen flow rate of 40 litres/min and manipulating the flows into and between the volumes until the model response had a close resemblance to the experimental results.

It was found during this analysis the transfer between the perfectly mixing sub-volumes was less significant than the nitrogen flow into each sub-volume.

Once this framework was established the model was compared to nitrogen flow rates between 20 and 100 litres/min. The results from the experiments show that the responses are close to those predicted by the model. **Figure 4.10(a)** illustrates the responses for a nitrogen flow rate of 20 litres/min. This was the least accurate, but errors are believed to be present due to instrumentation and inaccurate readings. The 60 litre/min response is typical of the higher flow rates with a very close response to those obtained from the experiments. **[See: Figure 4.10(b)]**

The results have confirmed the suggestion that accurate results can be obtained from specific lumped parameter models based on experimental data, and the engineer can consider options that he may believe would improve the system performance. It may be considered more important to reduce the oxygen concentration in the inner volume first and the outer volume is of secondary importance. If so it may be better to position the nitrogen inlet at the opposite end from the fan and fit a nozzle. This would ensure thorough mixing within the inner volume before the gases were drawn into the outer volume. A two element series model would probably describe the response, but all modifications and suggestions should be tested by experimentation to ensure validity.

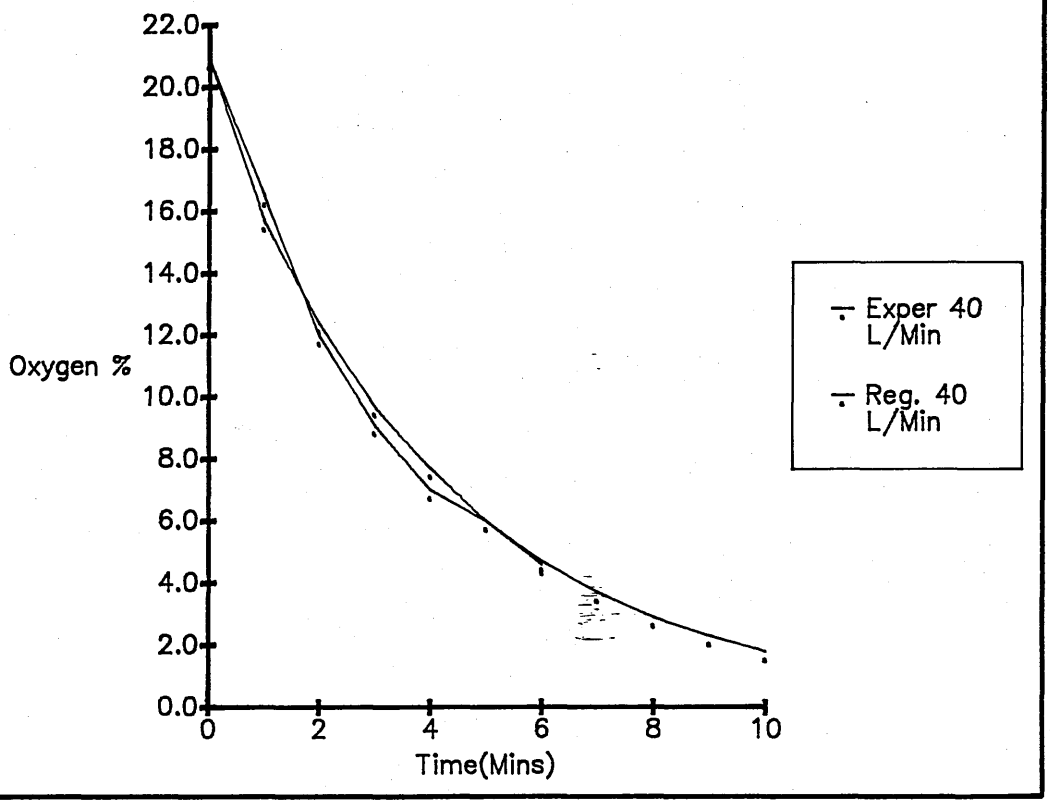
4.7 THE REGRESSION MODEL

The regression model has been suggested in Chapter 3 as the most accurate model if the information required is limited to how much nitrogen gas is required to achieve a specific oxygen concentration during a purge.

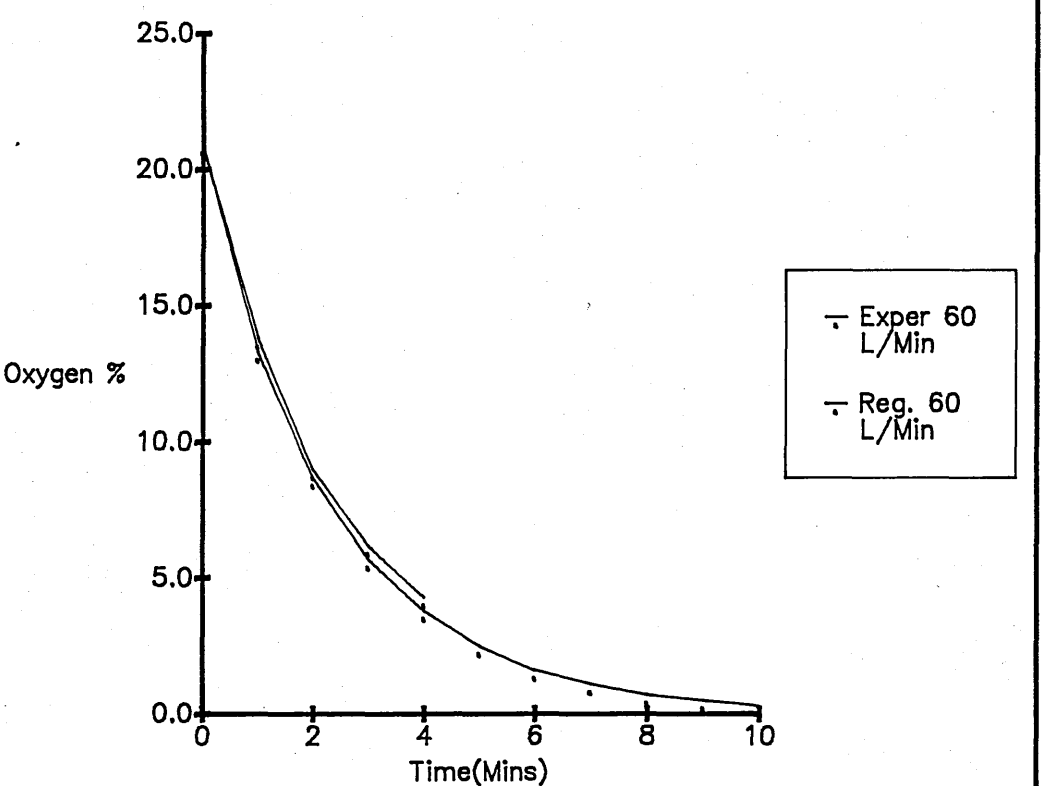
This type of model although accurate is specific not only to the system but also for a particular flow rate. The model is based on experimental data, monitoring the input and output while treating the system which connects the two as a "black box".

The relationship which connects the input and output is determined by a statistical technique known as linear regression.

Predicted Response Based on Regression Analysis
Compared to Actual Response of
The Model Autoclave
FIGURE 4.11(a)



Predicted Response Based on Regression Analysis
Compared to Actual Response of
The Model Autoclave
FIGURE 4.11(b)



In the case of the model autoclave the data for the system at various flow rates was taken and analysed as follows.

A graphical plot was made of the independent variable time against the dependant variable which was the natural logarithm of the oxygen concentration. This data was then processed by a computer programme to determine the regression equation which linked these two variables. A check for correlation and validity was made to ensure the accuracy of the equation. From this equation it is possible to determine the response at any moment in time.

Figures 4.11(a), (b) illustrate the results from the experiments and regression analysis.

4.8 CONCLUSIONS

The experiments on a simple clear volume have shown that even in a case where there appeared to be good grounds to assume a simple perfect mix model, considerable errors were in fact present. Although there were no physical barriers within the vessel the response suggested the vessel should be considered to have parallel sub-volumes.

Similar inaccuracies appeared when the perfect mix model was applied to the model autoclave and as this was the basis for the design of the inert gas plant for the real autoclave, which will be reviewed in Chapters 5 and 6, it is probable that there are errors present in the design.

These experiments have confirmed that the perfect mix model applied as if the vessel consisted of one simple volume is unsatisfactory and considerable inaccuracies can exist.

The alternative approach to the design using lumped parameter models has proved to be successful and could be recommended as a design method. Both during the initial design when used as a predictive model and when linked to experimental data. The advantage is that in addition to determining the amount of inert gas required it is possible to look at the probable gas movement within the vessel and assess where sampling points inlets and outlets should be positioned to the best advantage.

The regression model has proved to be a good method of determining the amount of gas required to purge a vessel or the time taken to achieve a particular atmosphere. The limitations are that the model can only be determined on the basis of accurate experimental results and it is specific to the application and flow rate.

Gas movement is important and it has been demonstrated that the amount of inert gas required to purge a complicated vessel can be reduced by the introduction of a fan. High mean velocities and turbulence create a more homogeneous mixture. The position of sampling points is less critical and any sample taken is more representative of the whole volume.

In general the experiments have confirmed the theory developed in Chapter 3 and selected aspects of the theory will be applied in the re-design of an inert gas plant for an autoclave.

CHAPTER 5

AUTOCLAVE – MODEL DEVELOPMENT

5.0 INTRODUCTION

It will be the aim in this Chapter to create a computer simulation model which will make it possible to accurately determine the size and configuration of an inert gas plant to satisfy the demands imposed by an autoclave.

5.1 THE AUTOCLAVE

The autoclave plays a vital role within an aerospace company where it is employed in the bonding of metal or composite materials to form components for the aerospace industry.

Experience has shown that under certain circumstances there is a risk of fire and fires have occurred. It is proposed to prevent this by creating an inert atmosphere within the vessel. A detailed analysis of the hazard in this particular case will be presented in the next Chapter **[See: Section 6.1]**

5.2 THE SYSTEM

The main element is a vessel which is 5.2m in diameter 12.8m long and it is capable of operating up to 17.2 bar at 673°K. The aircraft components are placed within the vessel on "tools" and are then subjected to a process cycle which raises the temperature and pressure thus completing a bonding process.

The high pressures are created by compressed air which is held in four gas reservoirs. These cylindrical vessels are 2.7m in diameter and 14.0m high. They are erected outside the factory, but adjacent to the autoclave vessel and are capable of operating up to 35.2 bar at 323°K.

The compressed air in the gas reservoirs is supplied from two air compressors each capable of providing air at 750m^3/hour at 273°K and 1.013bar. In normal operation only one compressor runs taking air in at atmospheric pressure and raising the pressure in the gas reservoirs to 32.0 bar.

High temperatures are created within the autoclave vessel by the recirculation of the air through an electric heater battery rated at 1.2MW. The gas movement is created by a fan at the rear of the vessel.

The existing autoclave-system with the main elements is illustrated in **Appendix C [See:FigureC.1]**. Currently inert gas is not used for any of the processes and an inert gas plant is not available although a design has been prepared and the costs estimated.

5.3 PROCESS SPECIFICATIONS

The production programme consists of 18 different process specifications (cures) with two additional cures for possible future inclusion.

The autoclave under review is known as the No5 Autoclave being the most recent addition in a group of five autoclaves of which it is the largest. Although it is capable of all the processes it only carries out certain cycles because for procedural reasons it is more convenient to allocate the other processes to other autoclaves.

Table 5.1 contains a summary of the information on the various cures.

Process Type	Temp °K	Press Bar	Theor Time Hrs	Estimate Time Hrs	Gas Consump Kg Moles	Relative Freq.
1	452	5.8	5.2	8.6	91283	0.23
2	452	3.1	4.9	8.4	51564	0.22
3	452	3.1	4.9	8.4	51564	0.05
4	452	2.3	4.9	8.4	38963	0.04
5	450	6.2	5.2	6.2	96576	0.07
6	400	3.1	3.5	5.8	47829	0.00
7	400	2.1	3.9	5.8	45180	0.00
8	452	0.7	2.8	7.6	11539	0.00
9	428	13.8	4.0	7.1	183587	0.01
10	428	9.6	3.1	6.7	130136	0.02
11	423	5.5	2.7	6.7	80329	0.00
12	423	1.7	2.6	6.7	27160	0.05
13	448	5.5	3.0	6.8	79227	0.00
14	448	3.4	3.9	6.8	55862	0.11
15	448	2.4	2.8	6.8	37226	0.02
16	448	1.2	4.7	6.8	20735	0.16
17	387	2.4	2.7	6.1	34796	0.00
18	387	3.1	2.8	6.1	44065	0.00
19	603	13.8	8.4			
20	603		12.8			

Process Specification Table
Source - Procedural Model 36 Week Forecast

TABLE 5.1

5.4 COMPUTATIONAL PROCESS MODEL

In order to estimate the amount of gas required for the various processes it was necessary to develop a computational model. This model is based upon a standard cure cycle which has the form illustrated in **Figure 5.1** and explained below. The model excludes the inert gas purging process which will be dealt with later **[See: Section 5.6]**

General Form:-

Although the time taken for each cure varies and the final pressure and temperatures may be different there is a general form. **[See: Figure 5.1]**

1). Pressure increases by 0.2 bar/min to the cure pressure.

2). Temperature increases to the specified temperature.between 1 and 3°C/min.

3). Conditions remain constant for a set period (Soak Period)

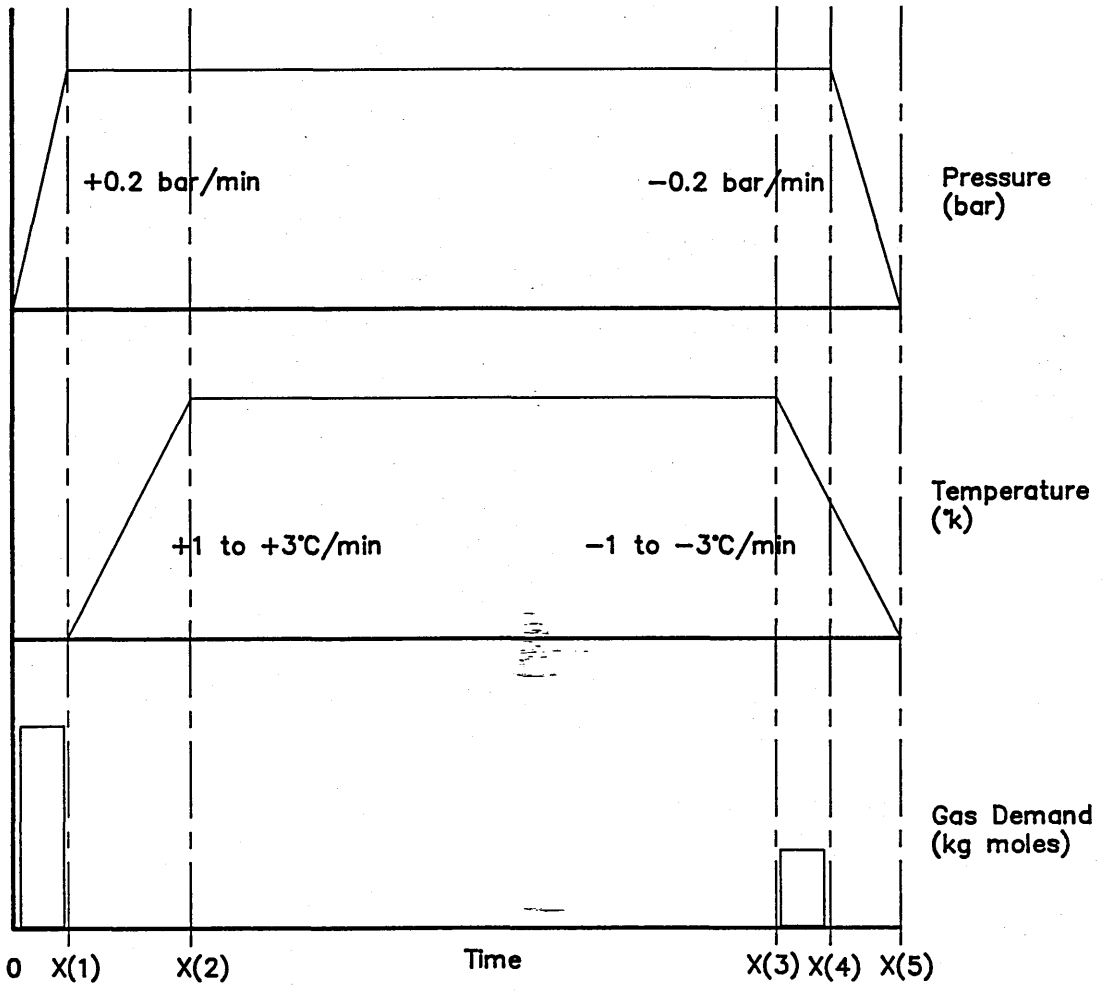
4). Temperature reduced to room temperature.

5). Pressure released.

5.4.2 Pressure - Rise/Fall:-

After the door on the autoclave is closed and all the initial tests completed the pressure in the vessel is increased at 0.2 bar/min to the specified cure pressure.

This 0.2 bar/min is set by the operators as a faster rise creates an unpleasant noise caused by the gas flow into the vessel.



0 to X(1) – Pressure Rise in Autoclave
X(1) to X(2) – Temperature Rise in Autoclave
X(2) to X(3) – Pressure and Temperature Remain Constant ("Soak Period")
X(3) to X(4) – Temperature Fall in Autoclave
X(4) to X(5) – Temperature and Pressure Fall

General Form of the Cure Cycle – Pressure, Temperature, Gas Demand (Excluding Purge and Flushing Phases)

FIGURE 5.1

The pressure is reduced at the end of the cure at 0.2 bar/min.

5.4.3 Temperature - Rise/Fall

The temperature rise and fall is specified in the process sheets and varies from 1 to 3°C/min. There is however a practical difficulty in achieving this.

Within the vessel there are a variety of tools and components with variations in mass, position and shape. These factors influence the rate of temperature rise of the tool and therefore not all components rise in unison. The operator makes a compensation for these lagging tools and he allows them to reach the necessary temperature before he starts the constant or soak period for the complete cure. The effect is the cures take considerably longer than the theoretical time and that is why, in Table 5.1, the estimated time is included. This is the time a cure will take based on experience of previous cures and there can be a considerable difference in the theoretical and estimated time. It should be pointed out this does not effect the demand for inert gas.

5.4.4 Kilogram - Mole

In order to simplify the analysis and allow for the fact that although air is presently used during cures it may be replaced by nitrogen the kilogram-mole unit is taken as the basis for the work.

5.4.5 Gas Consumption:-

Excluding any purge and flushing cycles the total gas consumption figures for the various processes is contained in Table 5.1.

The demand for gas during the process cycle falls into two areas. The initial pressurisation of the vessel at the start of the cure and at the end of a cure when the temperature is reduced, but the pressure is maintained. This is illustrated in **Figure 5.1.**

5.4.6 **Computer Programme:-**

The computer programme flow diagram is illustrated in general terms in **Figure 5.2.** The programme is written in Fortran 77 and run on a Digital VAX 750 Computer.

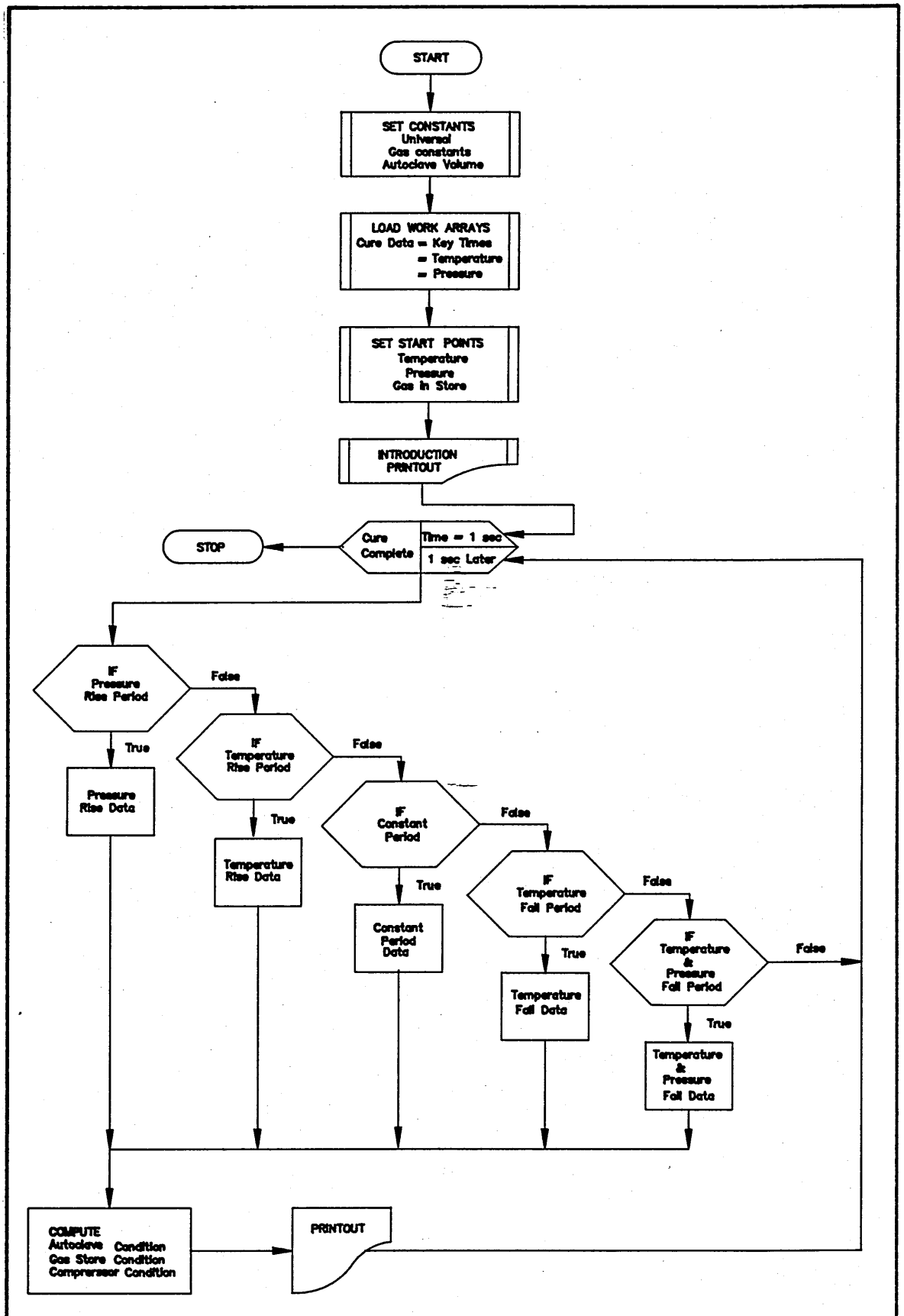
Gas Quantities:-

To calculate the variation in quantity of gas within the vessel during a cure it was necessary to derive a versatile equation with incremental changes in the two variables, temperature and pressure, and the resultant change in the quantity of gas within the vessel. The derivation of this equation is given in **Figure 5.3.**

5.5 PROCEDURAL MODEL

A procedural model has been developed by Short Brothers PLC to optimise the production methods and simulate future performance against estimated production requirements. The model predicts the operation of 4 autoclaves and the load sequence to support the production requirements.

This model has been validated against historical data and found to be acceptable.



Computational Process Model Flow Diagram
Figure 5.2

$$n = \frac{p.V}{R.T}$$

n = Kilogram moles
p = Pressure – (bar)
V = volume – constant – (m^3)
T = Temperature – (K)
R = Universal Gas Constant – (J/molk)
Δ = Small Incremental Change

Considering small incremental changes:–

$$(n+\Delta n) = \frac{(p+\Delta p)V}{R(T+\Delta T)}$$

Therefore:–

$$(n+\Delta n) = \frac{(p+\Delta p)V}{R(T+\Delta T)} = \frac{(p+\Delta p)V}{R.T}\left\{1+\frac{\Delta T}{T}\right\}^{-1}$$

The standard binomial expansion:–

$$(1+X)^N = 1 + \frac{N}{1}(X) + \frac{N(N-1)}{2!}(X)^2 + \ldots\ldots$$

Expanding:–

$$\left\{1+\frac{\Delta T}{T}\right\}^{-1} = \left[1 - \frac{\Delta T}{T} + \left\{\frac{\Delta T}{T}\right\}^2\right]$$

Therefore:–

$$(n+\Delta n) = \frac{(p+\Delta p)V}{R.T}\left[1 - \frac{\Delta T}{T} + \left\{\frac{\Delta T}{T}\right\}^2\right]$$

$$(n+\Delta n) = \frac{p.V}{R.T}\left\{1+\frac{\Delta p}{p}\right\}\left[1 - \frac{\Delta T}{T} + \left\{\frac{\Delta T}{T}\right\}^2\right]$$

Expanding and then working to the first order of small quantities:–

$$(n+\Delta n) = \left\{\frac{p.V}{R.T}\right\} - \left\{\frac{p.V}{R.T}\frac{\Delta T}{T}\right\} + \left\{\frac{p.V}{R.T}\frac{\Delta p}{p}\right\}$$

Therefore:–

$$\Delta n = \left\{\frac{V}{R.T}\right\}\Delta p - \left\{\frac{pV}{R.T^2}\right\}\Delta T \qquad \text{Equation 5.1}$$

Derivation of the Model Equation
FIGURE 5.3

The model is used to plan the day to day process sequence and to forecast any future production problems.

The model was run for 36 weeks and the results for the No.5 Autoclave analysed to determine the frequency of certain processes and the likely sequence. The relative frequency of occurrence of specific cures is tabulated in **Table 5.1.**

5.5.1 Data Analysis

The best production rate is approximately 14 cures per week.

From inspection of **Table 5.1** it can be shown that Type 1 and Type 2 processes occur most frequently.

The most individually demanding processes, from an inert gas consumption point of view, are those at high pressures such as a Type 9 at 13.8 bar.

Although a process may not appear in the relative frequency column of **Table 5.1** it does not mean that this cure is not carried out in the No.5 Autoclave. It has not appeared, or only infrequently in this simulation period.

At times it is necessary to repeat the start sequence for a cure as a tool can develop a fault which is repaired before the process is continued. This creates a greater demand for gas than appears from the list of successfully completed cures. This aspect is not adequately recorded at present, but has been allowed for in the process sequence that is used in the plant simulation model.

5.5.2 Process Classification

For the purposes of this study the processes have been considered in three groups based on the gas consumption.

1. Low Gas Demand 0 - 70000 kg moles

2. Medium Gas Demand 70000 - 120000 kg moles

3. High Gas Demand 120000 - 250000 kg moles

The sequence of processes that has been used in the plant simulation model when dealing with these three specific groups are tabulated in **Tables 5.2, 5.3** and **5.4.** The sequence is such as to be more demanding than the production rate at present to allow for any improvements in production, and repeated start sequences owing to excessive tool failures in the earlier stages of the cures.

Process Type	Temp °K	Press Bar	Gas Consump Kg Moles
2	452	3.1	51564
FAILURE			51564
2	452	3.1	51564
16	448	1.2	20735
FAILURE			55862
14	448	3.4	55862
3	452	3.1	51564
14	448	3.4	55862
FAILURE			51564
3	452	3.1	51564
15	448	2.4	37226
16	448	1.2	20735
15	448	2.4	37226

Low Gas Demand Process Cure Sequence

TABLE 5.2

PROCESS TYPE	TEMP DEG K	PRESS BAR	Gas CONSUMP KG MOLE
1	452	5.8	91283
FAILURE			91283
1	452	5.8	91283
5	450	6.2	96576
1	452	6.2	96576

Medium Gas Demand Process Sequence

TABLE 5.3

PROCESS TYPE	TEMP DEG K	PRESS BAR	Gas CONSUMP KG MOLE
FAILURE			130136
10	428	9.6	130136
FAILURE			183547
9	428	13.8	183547

HIGH GAS DEMAND PROCESS SEQUENCE

TABLE 5.4

5.5.3 Cure Failures

In Tables 5.2 to 5.4 it will be noted that in addition to the process sequence there are Failures. A failure is the situation where a cure starts, but is aborted at an early stage owing to a "tool failure" which is usually a failure in the tool component vacuum.

The tool is a sandwich consisting of a number of layers of material which are designed to ensure a successful bonding process. A vacuum is drawn over the sandwich sealed by a plastic bag. The complete package is placed within the autoclave where it is subjected to high pressures and temperatures. It is essential that the vacuum is held throughout the process and great care is taken to ensure that this is achieved. There are occasions when the vacuum fails and then the bonding of the component affected is deemed to have failed.

It is normal practice to raise the pressure first in the process cycle and it is during this time that a failure will usually occur. The operator can stop the process and release the pressure within the

vessel, enter the vessel and attempt to repair the fault.

A repair can take from 30 to 60 minutes.

This creates a greater demand for gas than indicated by the process sequence as there are two peak demands within a relatively short period of time. The first demand which is wasted followed by another demand some 30 to 60 minutes later. To make matters worse it is the high pressure cures with the high gas demand that most frequently experience failures.

There is considerable discussion as to the amount of gas wasted by these failures, but the simulation developed here has assumed the worst case. This is unlikely as from experience it can be shown that the faults appear within the first half of the pressurisation period, however the worst case is retained to show that even under these extreme circumstances the inert plant is capable of supplying the demand.

This area of cure failure requires further study as it can impose severe demands on the system and does have a major influence on the size of the inert gas plant.

5.6 PURGE CYCLE

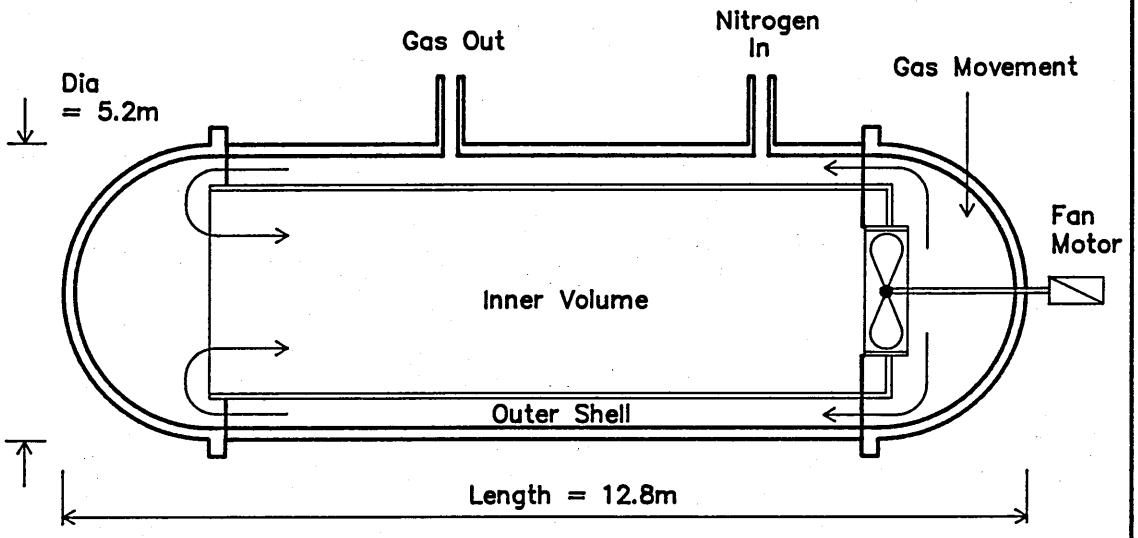
A recommendation has been made by American autoclave users that all processes should be carried out with an inert atmosphere. It is unlikely that this is actually required in all cases as is illustrated in the detailed hazard analysis of this particular autoclave in Chapter 6 **[See Section 6.1]**.

In order to develop a complete simulation model it is necessary to have a purge model available at the start of a cure to create the inert atmosphere, and a flushing sequence at the end of a cure. The flushing sequence is required to replace the inert gas with air so that the door of the vessel can be opened safely at the end of a cure.

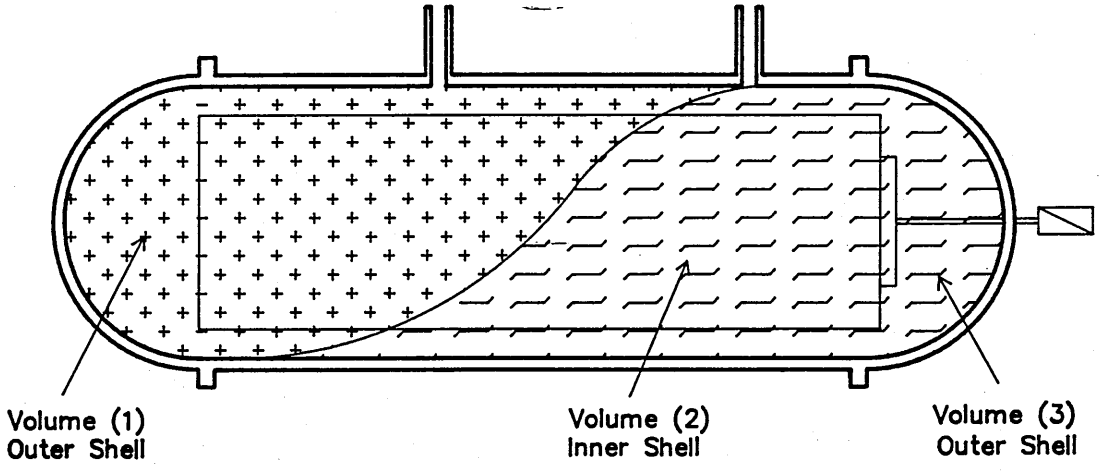
It is worth emphasising that although nitrogen is inert and non-toxic it is a dangerous gas at high concentrations and personnel can be overcome by the gas and lack of oxygen. Strict safety procedures must be set and adhered to, particularly in closed spaces. One of the main elements in the procedure is the removal of the nitrogen at the end of a cure by flushing the vessel with air.

The lumped parameter model that will be used as the basis for this purge model takes the form of three series volumes. [See:Figure 5.4]. This configuration differs from the model in Chapter 4, but as explained in Section 4.2 the model autoclave is only an approximate representation based on the information available at the time of construction which later proved to be inaccurate. On the basis of more accurate information it is felt that the three element series model is the best representation of the mechanisms within the autoclave vessel.

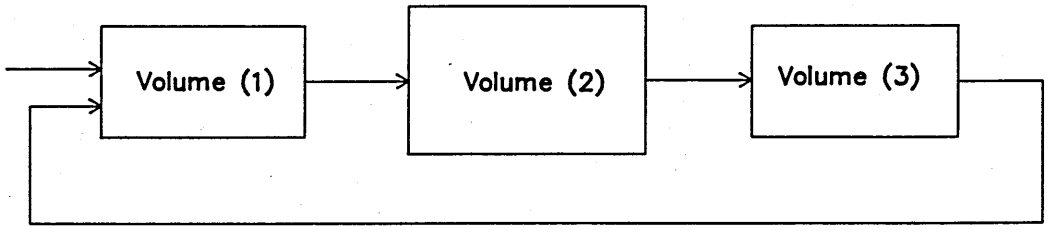
Sub-volume (1) and (3) represent the outer shell of the autoclave whilst sub-volume (2) represents the inner volume. The nitrogen gas is introduced to the outer shell into sub-volume (1) where it spreads and mixes under the influence of the gas movement created by the fan. It then enters the inner volume taken as sub-volume (2) and eventually passes to sub-volume (3) with the recirculating process continuing.



Autoclave Vessel Section



Autoclave Vessel Sub – Volumes



Autoclave Vessel Schematic

Actual Autoclave – 3 Element Series Purge Model

FIGURE 5.4

It is assumed that there will be effective mixing within these sub-volumes created by the fan and the obstructions within the vessel such as the heater battery, components and the corners of the vessel.

The area of specific interest is sub-volume (2) which is the inner vessel as it is here that combustion is likely to take place and the aim of the purging process is to create the inert atmosphere which will prevent or minimise the damage to components.

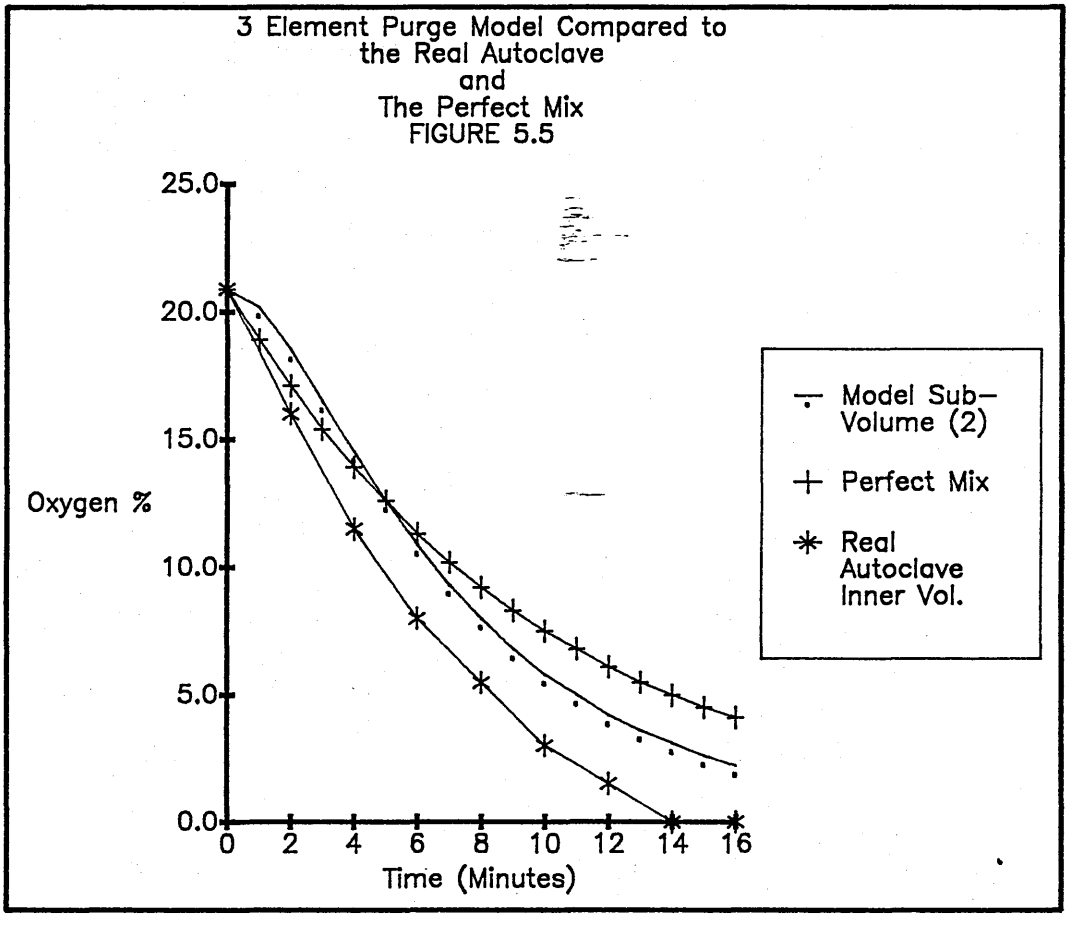
Experiments have been carried out on the real autoclave to test the validity of this three element model with the results shown in **Figure 5.5.** The simple perfect mix model response is also illustrated. As can be observed the simple perfect mix model gives a reasonable response for the first 5 minutes but after this period the error increases considerably and the 3 element model provides the best response compared to the actual system towards the end of the purge. It is this end period that is the most important as the purge process will normally aim to reduce the oxygen content to 0 - 5%.

The accuracy is best illustrated by an example:-

Assume it is necessary to reduce the oxygen to 5% prior to every cure by purging the vessel with nitrogen at a fixed flow rate - how long would it take ? :-

Real Autoclave	8 minutes
3 Element Model	11 minutes
Simple Model	14 minutes

It is obvious from these results that the multiple lumped parameter model can provide a greater degree of accuracy and therefore it will be applied as the


3 Element Purge Model Compared to
the Real Autoclave
and
The Perfect Mix
FIGURE 5.5
25.0
20.0
15.0
10.0
5.0
0.0
Oxygen %
0
2
4
6
8
10
12
14
16
Time (Minutes)
Model Sub–
Volume (2)
Perfect Mix
Real
Autoclave
Inner Vol.

purging and flushing model in the complete computer simulation.

5.7 SIMULATION MODEL

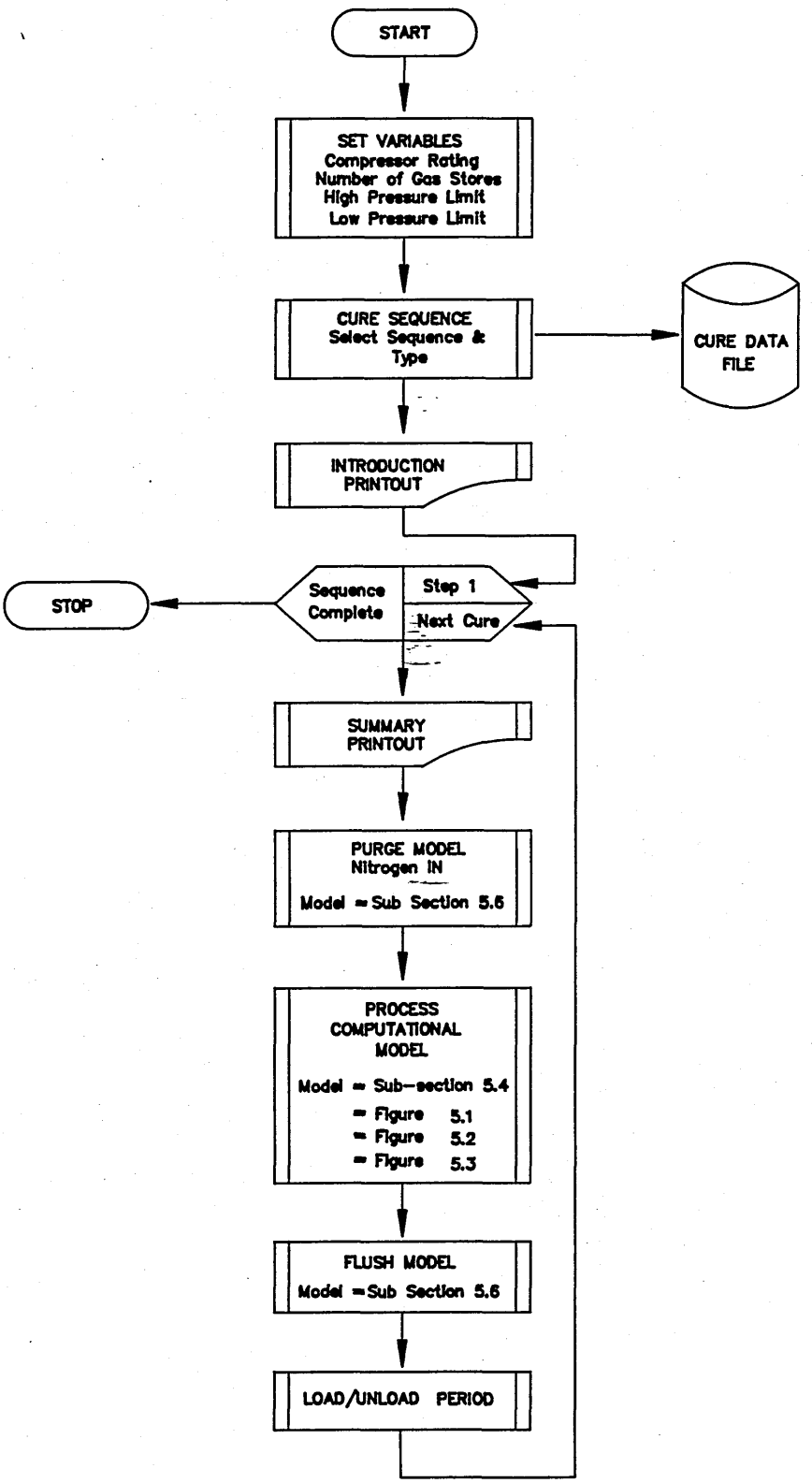
At this point it is possible to develop a computer simulation model for the whole autoclave air and gas supply system. This will allow the engineer to vary the system. He may now determine the optimum inert gas configuration that will satisfy the autoclave demand for inert gas in the most economical manner.

The simulation model consists of the following elements:-

(1) Procedural model for the sequence of processes.
(2) Purge model.
(3) Process computational model.
(4) Subroutine for the compressed gas vessels.
(5) Subroutine for the air compressors.
(6) Subroutine for the nitrogen buffer stores.
(7) Subroutine for the nitrogen production plant.

The general flow diagram for the simulation model is illustrated in **Figure 5.6.** The programme uses the models that have been discussed previously in this chapter namely, the process computational model, the procedural model and the purge model.

The procedural model determines the most likely series of cures with the selection made on the basis of the most arduous schedule for the process groups. The three groups are defined as low, medium and high which reflect the relative gas consumption of the various cures.



Autoclave Simulation Model Flow Diagram

FIGURE 5.6

The purge model calculates the quantity of inert gas used in purging the autoclave at the start of a cure and the amount of air required to flush the vessel clear of inert gas at the end of a cure cycle.

The process computational model calculates the amount of gas within the autoclave and how much gas is used during each cure.

The subroutines for the plant components determine the operational status of the various elements and their input to the system.

5.7 1 Advantages

With the simulation model it is possible to consider more facets of the installation than is possible by manual techniques and the effect on other components in the system can be assessed.

This model has been designed so that the following parameters can be treated as variables and are set prior to each simulation run:-

(1) Compressor rating
(2) Number of Gas Stores
(3) High and Low Pressure Control Limits
(4) Type and Sequence of Cures

5.7.2 Validation of the Simulation Model

It is important to validate any model and this was possible in this case by monitoring the plant operation during normal production runs. It was not possible to obtain the release of the autoclave from normal production in order to check the performance during all cures, therefore the validation checks

had to be limited to the sequence of cures set by production.

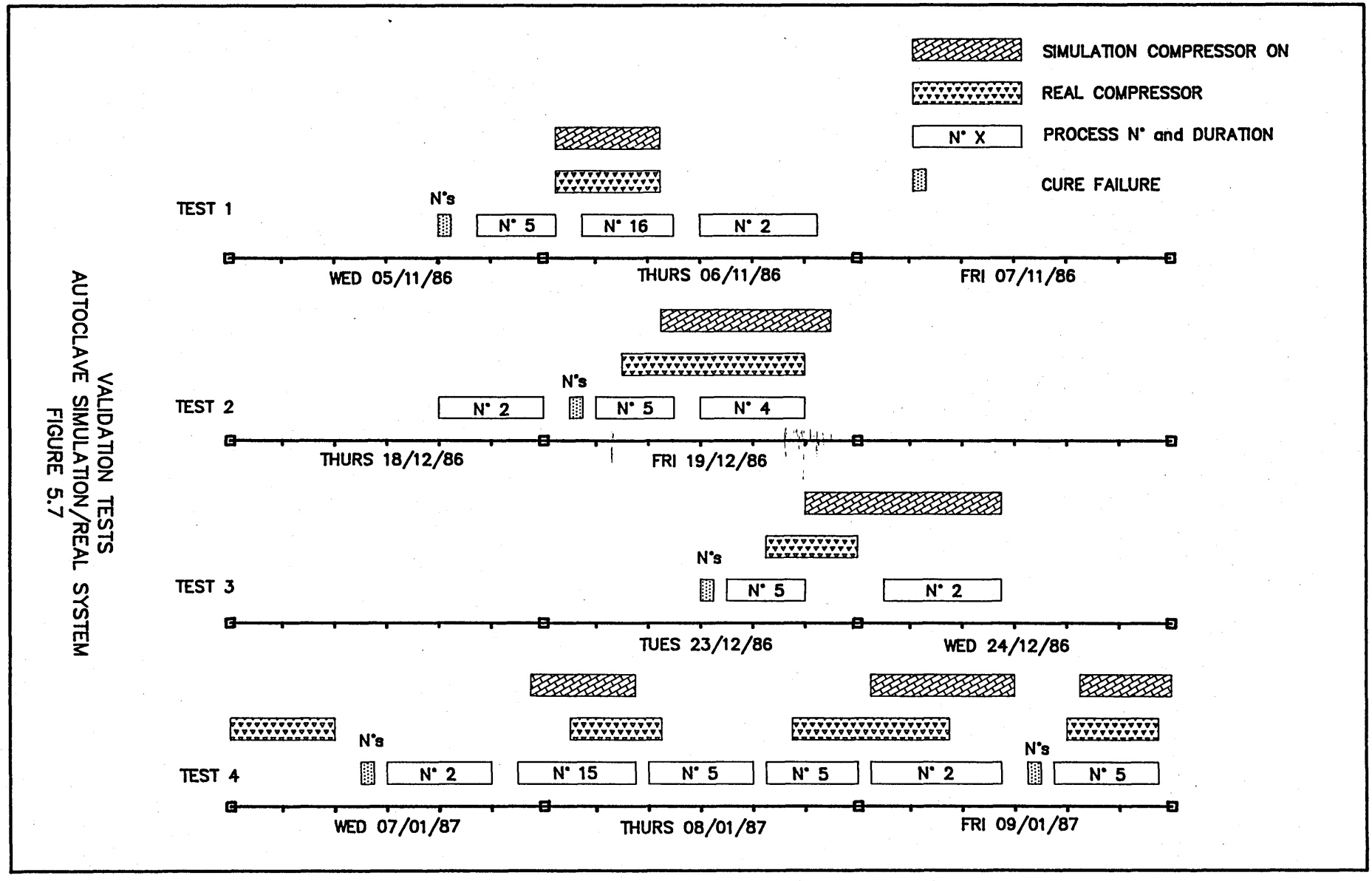
An event recorder was connected to the existing compressor to record when the compressor was ON or OFF. The autoclave operators kept detailed notes on the type of cures, any failures which meant a repeated start sequence and the pressure in the reservoirs at the start and end of the cures.

There was sufficient data returned for four simulation runs with the model parameters set to the conditions that prevailed during the data collection period. The results from these simulations are compared to the real behaviour in **Figure 5.7.**

The nitrogen plant and buffer store subroutines were excluded from the computer simulation validation tests as they are not part of the current system. It is felt that this does not unduly effect the validation tests as the main elements in the model are tested. Items (1) to (5) in **Section 5.7.**

The results show a close similarity with regard to the total compressor run time although the start and stop times did not exactly coincide. The reason for this variation became evident on closer examination of the control settings and the operational logic.

The compressor is controlled by two pressure switches connected to the gas reservoirs. One controls the high pressure limit and the other the low pressure limit. These devices although accurate enough for their normal function were not as accurate as the limits set within the computer simulation. When the low pressure limit is triggered the compressor starts and continues to run until the high pressure limit is reached. After a demand for gas the pressure within the gas



VALIDATION TESTS
AUTOCLAVE SIMULATION/REAL SYSTEM
FIGURE 5.7

reservoirs may have fallen to 20.01 bar, at which point the compressors would start on the real system, but the simulation compressors would not start until the pressure within the simulated gas reservoirs reached 19.99 bar.

This anomaly does not unduly effect the accuracy of the model and in fact has brought to light other factors which could considerably improve the operation of the autoclave if a more sophisticated control system was introduced.

5.8 CONCLUSIONS

The aim of this chapter was to create a computer simulation model that would accurately reflect the performance of the autoclave and gas supply.

This has been achieved by the development of models for three main areas. The gas consumption during the process cycle, the probable sequence of cures and the purging process. Each model has been compared to historical or experimental data and found to be valid.

To allow for a variation in the size of compressors etc. and the control logic the simulation model has been written with certain key elements as variables. These can be set at the start of each simulation run.

The simulation model has been validated against real data and found to be successful and accurate. It will now be used in the detailed analysis of the autoclave system.

CHAPTER 6

AUTOCLAVE — AN APPLICATION

6.0 INTRODUCTION

The autoclave presently uses air as the compressed gas medium to increase the pressure and provide the means of heat transfer to the components.

It has been suggested that in all cases the air should be replaced by nitrogen to prevent the risk of combustion.

Budget costs and an initial design have been prepared for this inert gas plant to support production on the basis that all cures require inert gas. It had been assumed the purging process response conformed to a perfect mix with the autoclave treated as a simple clear volume and the demand for inert gas calculated on a weekly basis.

As there are a number of errors in the approach taken the inert gas plant will be re-designed using the techniques developed in this thesis. The effects on the original estimates of a capital cost of £500,000 and a running cost of £160,000/annum. will be assessed.

6.1 HAZARD ANALYSIS

An opinion has been given by a number of autoclave users that a hazard exists during all processes.

This is inaccurate as the degree of hazard varies with the increase in pressure and the rise in

temperature. As the pressure increases so the system moves into the sphere of an oxygen enriched atmosphere and the normal characteristics of materials alter. **[See:Chapter 2]**

The fact is that not all processes in the autoclave are at high pressures as is shown in **Table 5.1** where the frequency of occurrences of the various cures is tabulated.

This analysis has been divided into three types of processes in accordance with the classifications set in Chapter 5 **[See:Sub-section 5.5.2]** namely, low, medium and high gas demand. The demand for gas is directly related to the process pressure which is linked to the degree of risk due to an oxygen enriched atmosphere if air is used.

6.1.1 Flammable Materials in the Autoclave

There are no flammable liquids, vapours or gases within the autoclave under normal circumstances.

Factory dust may accumulate within the vessel, but there is no specific flammable dust in sufficient concentration to create a hazard. **[See:Sub-section 2.4.3]**. As a precautionary measure however, the vessel should be cleaned periodically to prevent the excessive accumulation of dust. The dust is not a problem in itself, but may add to a fire if one started by some other means.

The only solids within the vessel which are a potential fuel are the items forming the tools, in particular the sheeting which seals the component. Simple tests were conducted in air at atmospheric pressure with samples of the materials ignited with a flame produced by a wood taper and the material

was found to burn with a self sustained diffusion flame. It can therefore be expected to burn with accelerated combustion in an oxygen enriched atmosphere. **[See:Section 2.1 and Sub-Section 2.3.2].** If an ignition source was present with sufficient energy then combustion could take place, particularly at the higher pressures where the effect of an oxygen enriched atmosphere comes into play.
[See:Chapter 2.]

It is strongly recommended that detailed tests are carried out on these materials to determine their exact flammability characteristics under various conditions. In particular the hot plate ignition temperature under various environmental pressures. **[See:Sub-Section 2.3.2]**

6.1.2 Low Demand Cures

In the 36 week period forecast, on which this study is based, 65% of the processes are in the group defined as Low Demand Cures. **[See:Sub-section 5.5.2]**

The maximum temperature during these cures is 452°K and the highest pressure is 3.4 bar.

There have been few problems with these cures over the years when air has been used and the temperatures are well outside the conditions for autoignition for the materials in use.

It is true that an increase in pressure changes the flammable characteristics of a material as described in Chapter 2, but even so the conditions are within safe limits for normal circumstances.

This is supported by **Table 2.2** which contains the results from hot plate ignition temperatures, for more readily flammable materials, under various pressures. Even under pressures of 4 bar the minimum ignition temperature for materials such as cotton sheeting or paper drapes is in excess of 600°K.

It is true however, that high ignition energies could be created by a static discharge or illegal flammable material left on a tool, such as an oily rag, which could ignite. These conditions must be considered.

The problem of static can be considerably reduced with adequate bonding and the ample spacing between component, but illegal flammable material can only be overcome by good working practices and supervision.

As there is a chance that a fire may start, although the probability is low, some method of dealing with it should be available in order to restrict the damage. Inert gas in a flame extinguishing role in the event of a fire being detected would be advisable.

Relatively low levels of inert gas would be acceptable as the fire protection strategy would be to release the vessel pressure and keep the fan running. The nitrogen would then quickly mix within the vessel under atmospheric pressure and the typical fire extinguishing data in **Chapter 1** would be valid.

6.1.3. Medium Demand Cures

In this study 30% of the processes were within the group defined as Medium Demand Cures. **[See:Sub-section 5.5.2]**

The maximum temperature is 452°K and the highest pressure is 6.2 bar.

Although the temperatures are still relatively low compared to the autoignition temperatures of the materials, the increase in pressure tends to reduce the hot plate ignition temperature and the ignition energy required to ignite the material. **[See:Sub-Section 2.3.2]**

These cures are currently carried out using air as the medium with generally few problems, but there have been instances of fire.

The same problems exists with these processes as with the low demand cures in that static or illegal combustible materials may provide the source for ignition. The danger increases in this case as the higher pressures tend to reduce the required ignition energies. **[See:Chapter 2]**

At this point a decision has to be made on the economics of providing an inert atmosphere as compared to the loss of one or more components within the vessel due to a fire. This decision should be made in the light of current experience with the process carried out using compressed air, but with an inert gas extinguishing system.

The recommendation made here is that although experimental work is required to determine the behaviour of the materials under these conditions a partial inert atmosphere should be created within

the vessel to prevent the propagation of any diffusion flame or the development of a premixed flame.

Data is not available on the desired quality of this partial inert atmosphere, but on the basis of the work by Hirst **[See:Table 1.3]** and the effect of nitrogen on the combustion of paper which is more flammable **[See:Figure 2.1 and Table 2.2]**, a 70% nitrogen in air atmosphere would prevent combustion. If under abnormal circumstances combustion were to take place even under these conditions the nitrogen level could be raised to 90% in air in a relatively short space of time.

6.1.4 High Demand Cures

Only 5% of the cures are within this group, but it is the most demanding and there is a definite risk of fire. If air is used the atmosphere is well into the oxygen enriched region. Relatively low ignition energies could initiate combustion with high energies starting secondary combustion of other materials. **[See:Chapter 2]**

It is recommended that all cures in this group use nitrogen gas at a concentration of 90% in air.

6. 2 THE SELECTION OF NITROGEN AS THE INERT GAS

Nitrogen was selected as the inert gas for the application because:

(1) It is non toxic and safe within limits. **[See: Section 5.6]**

(2) It is inert and will remain stable during these processes.

(3) It is readily available

(4) It is less expensive than other available inert gases.

The gas can be supplied to the system by a number of methods:

(1) Bulk delivery of liquid nitrogen from a Gas Manufacturer. Bulk liquid storage with the nitrogen vaporised and compressed to supply the system as required.

(2) Manufactured on site by absorption, from the atmosphere.

(3) Manufactured on site by fractional distillation.

(4) Gas combustion Generator.

6.3 THE SIMULATION & ANALYSIS

The aim of this work is to determine the most cost effective installation to match the demand created by the processes and production schedule with on-site production based on nitrogen from a fractional

distillation plant. This was selected as it was the basis for the inert gas plant as originally designed and it is proposed to compare the results produced by the design methods put forward in this thesis with those obtain by the original design methods.

There were four cases considered namely:-

(1) The existing system using compressed air.

(2) Low Demand cures using compressed air.

(3) Medium Demand cures using air/nitrogen atmospheres with a 70% nitrogen concentration.

(4) High Demand cures using air/nitrogen atmospheres with a 90% nitrogen concentration.

For each of these cases there was a detailed analysis carried out using the autoclave computer simulation model developed in Chapter 5.

The aim was to obtain for each case an equation which linked the size of the key components such as gas vessels, compressors etc. to the total capital cost of the plant with the aim to manipulate the rating and capacity of these key components to reduce the capital cost to a minimum.

This was possible by treating the system components as variables and changing them in a random manner, but ensuring that no matter which configuration was used it was capable of meeting the process gas demand. This was checked by running the simulation model and ensuring that at no point did the system fail to supply gas.

The variables were:-

y = Total Capital Cost (Estimated)
x(1) = Gas Reservoir Volume
x(2) = Compressor Rating
X(3) = Nitrogen Plant Rating
x(4) = Buffer Store Volume.
[x(1) to x(4) converted to equivalent costs]

For each of the four cases **[See: Section 6.3 Para.2]** a series of simulation runs were carried out on the computer. Data was gathered for each variable and a group of equations produced for each of the four cases with the form:-

$$y(i) = x(1)i + x(2)i + x(3)i + x(4)i \quad \textbf{[Eq. 6.1]}$$

The equations were processed by a computer running a programme for multiple linear regression. The programme in addition to determining the coefficient for each variable checked the validity of the equation by variance analysis. The result of this analysis was a series of equations for each of the four cases taking the form:-

$$y = ax(1) + bx(2) + cx(3) + dx(4) \quad \textbf{[Eq. 6.2]}$$

In order to determine the optimum size of the various elements to meet the process demands, but at the minimum cost the equations were processed by a computer programme written for the solution of equations by linear programming.

The result of this analysis was a set of four equations which indicated the optimum size of the key components for each of the four cases. The components such as compressors, gas storage vessels and nitrogen plant etc. were sized on the basis of the nearest available size and the autoclave plant simulation model was run again for each of the four

cases to ensure that the combination met the demand even under the most rigourous conditions.

In addition to running the plant simulation model through the production programmes set in Tables 5.2 to 5.4, the systems were tested to see how they would behave under fault conditions such as compressor failure, and how long would it take to bring the system up to normal operation from an empty gas reservoir condition.

6.4 THE EXISTING SYSTEM

One of the interesting facts that came to light was the existing compressor arrangement is oversized. The existing cure sequence could be met with an air compressor with a rating of 500m^3/hour rather than the existing 750m^3/hour. In addition with minor modifications to the control system this could be further reduced to 300m^3/hour. The modifications would allow the compressor to take advantage of the long periods when there is no demand with the compressor OFF, but the gas reservoirs are sitting at two thirds the maximum pressure. During the quiet periods between loads and during the "soak periods" the compressor could be started and given the opportunity to refill the reservoirs. The effect on the system is a reduction in the size of the compressor required to meet the demand.

6.5 LOW DEMAND CURES

As indicated by the hazard analysis [See:Section 6.1.2] an inert atmosphere is not required as the risk of combustion is low therefore the design is on the basis of air as the compressed gas. It is recommended however, that a nitrogen extinguishing

system should be available in the event of combustion.

The result of the analysis suggests the optimum plant configuration to meet the process requirements would be:-

Two - Gas Stores for Compressed Air (83m^3 each)
One - Air Compressor (500 Nm^3/hr)

(N = @ 273°K and 1.013 bar)

The nitrogen for fire extinguishing would be supplied from the system that will support the medium and high gas demand cures.

6.6 MEDIUM DEMAND CURES

Although there is an increased risk it is not high and therefore the recommendation made here is that an atmosphere with 70% nitrogen in air will be sufficient to prevent combustion.

The result of the analysis suggests the optimum plant configuration to meet the process requirements would be:-

Two - Gas Stores for Nitrogen (83m^3 each)
One - Nitrogen Compressor (500 Nm^3/hr)
One - Nitrogen Plant (500 Nm^3/hr)

(N = @ 273°K and 1.013 bar)

The compressed air will be provided from the system that supplies the low gas demand cures.

6.7 HIGH DEMAND CURES

These cures require an atmosphere with a nitrogen concentration of 90% in air. To support this the analysis suggests the following configuration:-

Four - Nitrogen Gas Stores (83m^3 each)
One - Nitrogen Compressor (500 Nm^3/hr)
One - Nitrogen Plant (500 Nm3/hr)

(N = @ 273°K and 1.013 bar)

The flushing air required at the end of a cure to remove the nitrogen will be supplied from the system that supplies the low demand cures.

6.8 THE OPTIMUM SYSTEM

At this point the optimum solutions to each of the four cases have to be brought together to produce the optimum practical plant configuration that will meet all the process demands and safety requirements.

The proposed system that would be most cost effective and capable of all the processes set in the procedural model **[See:Table 5.1]** is illustrated in Figure C.2 in Appendix C.

There would be six gas storage vessels, two compressors and one nitrogen plant.

Gas Stores - 2 for compressed air to supply the low demand cures and provide the air to quickly flush the autoclave free of nitrogen at the end of a nitrogen cure.

- 2 for nitrogen to supply the medium demand cures

- 2 for nitrogen buffer stores to hold the reserve and allow the nitrogen plant to run efficiently. Some nitrogen plant will not operate well for short periods.

These two buffer stores would be connected to the other nitrogen stores to provide the peak demand for the high demand cures.

The buffer stores would supply the nitrogen for the extinguishing system which would operate during low demand cures if a fire was detected.

6.9 THE ORIGINAL INERT GAS PLANT DESIGN

The original design suggested that the existing four air storage vessels should be converted to nitrogen storage and the existing two 750 Nm3/hr compressors should be modified so that they would take a nitrogen supply from the nitrogen plant. The result was that the nitrogen plant had to be capable of supplying gas at 1500 Nm^3/hour.
(N = @ 273°K and 1.013 bar)

These decisions were made on the basis of:-

(1) A high nitrogen concentration during all cures (90 - 95%)

(2) A 20 minute purge time at the start of each cure

(3) The assessment of the consumption on a weekly basis and assuming the highest possible gas demand.

The design was prepared on this basis and an estimate of the cost made which indicated the capital cost would be £500,000 with an annual running cost of £160,000.

6.10 ERRORS

There are a number of errors in the original design of the system.

(1) High levels of inert gas are not required for every process, 65% of the normal load will operate safely with compressed air, 30% with 70% nitrogen and 5% with 90% nitrogen.

(2) The purge time is not 20 minutes but 14 minutes and therefore the purge cycle consumption is considerably less than anticipated.

(3) The gas consumption was calculated on a weekly basis. This failed to take into account the peak demand imposed by the processes which consisted of sudden loads followed by long periods with no demand for gas with inadequate attention given to the gas production system and control system.

(4) No accurate basis for the load schedule was determined and the worst case was assumed which from a factory production viewpoint is impossible to achieve. Closer consultation with the Production Engineers would have produced a more realistic production schedule

6.11 FINANCIAL ADVANTAGES

If the design methods employed here had been applied in the original autoclave design the results would have shown that a 500 Nm^3/hour air compressor would have been sufficient rather than those selected at 750 Nm^3/hour. The estimated savings would have been £20,000 which excludes the reduced maintenance and running costs.

The inert gas plant configuration illustrated in **Figure C.2 [See: Appendix C]** would result in a saving of £60,000 on capital outlay and an estimated reduction in running costs of £50,000/annum. These prices are based on figures that were current at the time of the original design which was in 1982.

6.12 CONCLUSIONS

Failure to appreciate the detailed aspects of such a system and attempting to design by manual steady state methods will lead to incorrectly sized plant.

By using alternative methods such as simple and multiple linear regression linked to linear programming and computer simulation it is possible to increase the accuracy of a design and optimise the installation.

The aim is to meet the demand for inert gas and create the required inert atmosphere at the minimum cost. This can be achieved with confidence using the design methods described here with the major benefits of reducing the capital and running costs.

CHAPTER 7

DISCUSSION and CONCLUSIONS

The main aim of the work was to consider the application of inert atmospheres for the control of combustion in vessels and a number of important points have become apparent.

(1) There is little data available on the quality of inert atmosphere required for the wide variety of industrial processes, but from examining the data available and the mechanisms involved it is obvious that it is not necessary to create high concentrations of inert gas in every case.

(2) The combustion characteristics of materials can change radically in modified atmospheres. An oxygen enriched atmosphere is one form of modified atmosphere which arises on a frequent basis whenever oxygen or compressed air is utilised or stored. The general effect is to increase the combustion hazard.When considering the data that is available care must be taken as frequently it is in relation to ambient temperature and pressure.

(3) There are a wide range of factors to consider when determining the quality of inert atmosphere required to control combustion. To obtain accurate data it is necessary to conduct experiments for the particular application and examine closely past experience with the process.

Once the quality of the inert atmosphere has been determined the quantity of gas required to create this atmosphere and its method of introduction has to be determined. The conclusion reached here is that the multiple lumped parameter modelling technique provides a good practical approach to this analysis with a reasonable degree of accuracy. In addition to determining the amount of inert gas to create a specific atmosphere it gives the opportunity of an insight to likely gas movements within the vessel indicating where sampling points should be positioned, inlet positions and the desired mixing rate between sub-volumes.

Such models should be supported wherever possible with experiments on similar plant or scale models to ensure the suggested sub-volume arrangement is valid.

When designing an inert gas plant there are other factors to consider in addition to the quality of atmosphere and the quantity of gas to create the specific atmosphere such as:-

(1) How frequently will the demand for inert gas be imposed on the system ?

(2) How much gas should be held in reserve ?

(3) By what means will the inert gas be obtained ie. Bulk Delivery or On-site Production ?

(4) What is the best inert gas plant configuration to support production at the minimum capital and running cost ?

Currently, in plant engineering, these factors are considered using manual design techniques combined with experience and intuitive decisions to determine

the optimum solution. A more balanced and rigourous approach is to develop a computer simulation model which allows an engineer to evaluate the options.

This approach has been developed to examine a previous design based on manual methods for an inert gas plant for an autoclave. The simulation model has proved to be accurate and highlighted a number of points which could improve the operation of the autoclave. When the results from the simulation have been analysed by using multiple linear regression and linear programming to determine the optimum solution it has been shown that the inert gas plant, as originally designed, was oversized.

The solution proposed in this thesis deals with the hazard and yet reduces the proposed capital outlay by £60,000 (12%) and the estimated annual running cost by £50,000 (31%).

This type of simulation analysis could be applied to a wide variety of applications in plant engineering. The models developed here were complicated and time consuming to produce and verify as the programmes were written in Fortran or Basic. However, the results have shown that the work is worthwhile.The procedure will become less difficult in the future with the development of suitable simulation software.

In the case of the real autoclave the lumped parameter purging and flushing model did provide a more accurate assessment of the amount of gas required for the process, but the major savings were achieved through the development of an accurate procedural model and the detailed analysis of the results produced by the plant simulation model.

APPENDIX A

RESULTS

NOTES:-

1. Sample taken from outlet of the vessel.
2. Vessel Dimensions - dia = 0.61m, len = 1.00m.
3. Len/Dia. Ratio 1:1.6

FLOW L/Min	20	40	80
TIME SECS		% OXYGEN	
0	20.9	20.9	20.9
30	20.9	19.5	18.7
60	20.9	18.1	15.0
90	20.3	16.2	12.2
120	18.9	14.4	9.8
150	17.0	12.9	7.8
180	15.9	11.5	6.3
210	15.3	10.4	5.0
240	14.5	9.6	4.0
270	14.2	8.7	
300	13.4	7.7	
330	12.3	6.7	
360	11.0	6.1	
390	11.0	5.4	
420	10.3	4.9	
450	9.4	4.3	

Typical Results from Experiments on a Simple Volume

TABLE A.I

REGRESSION ANALYSIS FOR 20 LITRES/MIN

TIME	LN % OXYGEN
0.000	3.040
30.000	3.040
60.000	3.040
90.000	3.010
120.000	2.940
150.000	2.830
180.000	2.770
210.000	2.730
240.000	2.670
270.000	2.650
300.000	2.600
330.000	2.510
360.000	2.400
390.000	2.400
420.000	2.330
450.000	2.240

MEAN VAR X	=	225.000	MEAN VAR Y	=	2.700
SD VAR X	=	142.829	SD VAR Y	=	0.271
CORRELATION	=	-0.991			

REG COEFF	=	-0.002	REG CONST	=	3.123
SE OF COEFF	=	0.000	SE OF CONST	=	0.018
T-VALUE	=	-27.269	T-VALUE	=	171.562

REGRESSION

SOURCE	SUM OF SQUARES	DF	MEAN SQUARE	F-VALUE
DUE TO REG	1.08	1.	1.08	743.57
ABOUT REG	0.02	14.	0.00	
TOTAL	1.10	15.		

Typical Regression Analysis with Results from Experiments on a Simple Volume

TABLE A.2

APPENDIX B

RESULTS

NOTES:

1. Nitrogen injected into the centre of the vessel.
2. Recirculation fan ON.
3. Sample taken at outlet.

FLOW L/Min	20	35	40	60	80
TIME Secs.	% OXYGEN				
0	20.9	20.9	20.9	20.9	20.9
30	19.5	19.3		16.7	16.8
60	18.2	17.3	16.5	13.8	12.6
90	16.9	15.2	14.0	11.2	9.4
120	15.6	13.4	12.0	9.0	7.0
150	14.6	12.0	10.0	7.2	5.2
180	13.6	10.6	9.1	6.2	3.8
210	12.6	9.2	8.1	5.3	
240	11.7	8.1	7.0	4.3	
270	10.9	7.2		3.5	
300	10.2	6.6	6.0		
330	9.5	5.6	5.2		
360	8.8		4.6		
390	7.6	4.9	4.0		
420		4.4			
450					

Typical Results from Experiments on a Model Autoclave
Recirculation Fan ON

TABLE B.I

REGRESSION ANALYSIS FOR 20 LITRES/MIN

TIME	LN % OXYGEN
0.000	3.040
30.000	2.970
60.000	2.900
90.000	2.830
120.000	2.750
150.000	2.680
180.000	2.610
210.000	2.530
240.000	2.460
270.000	2.390
300.000	2.320
330.000	2.250
360.000	2.170
390.000	2.020

MEAN VAR X	= 195.000	MEAN VAR Y	= 2.566
SD VAR X	= 125.499	SD VAR Y	= 0.313
CORRELATION	= -0.998		
REG COEFF	= -0.002	REG CONST	= 3.052
SE OF COEFF	= 0.000	SE OF CONST	= 0.010
T - VALUE	= -55.706	T-VALUE	= 297.264

REGRESSION TABLE

SOURCE	SUM OF SQUARES	DF	MEAN SQUARE	F-VALUE
DUE TO REG	1.27	1.	1.27	3103.12
ABOUT REG	0.00	12.	0.00	
TOTAL	1.28	13.		

Regression Analysis of Results from Experiments on a Model Autoclave (Fan ON)

TABLE B.2

RESULTS

NOTES:

1. Nitrogen injected into the centre of the vessel.
2. Recirculation fan OFF.
3. Sample taken from the outlet of the vessel.

FLOW L/MIN	20	40	80
TIME		% OXYGEN	
0	20.9	20.9	20.9
30	20.9	20.4	18.1
60	20.0	18.0	12.7
90	19.6	14.3	8.9
120	17.8	13.1	6.3
150	16.0	11.7	4.5
180	15.9		3.1
210	14.6	8.6	
240	14.2	7.4	
270	13.1	6.4	
300	12.3	5.7	
330	11.1	4.8	
360	10.2	4.4	
390	9.8	3.6	
420	8.6		
450	8.0		

Typical Results from Experiments on a Model Autoclave
Recirculation Fan OFF

TABLE B.3

REGRESSION ANALYSIS FOR 20 LITRES/MIN

TIME	LN % OXYGEN
0.000	3.090
30.000	3.090
60.000	3.040
90.000	2.970
120.000	2.880
150.000	2.770
180.000	2.760
210.000	2.680
240.000	2.650
270.000	2.570
300.000	2.510
330.000	2.410
360.000	2.320
390.000	2.280
420.000	2.150
450.000	2.080

MEAN VAR X	=	225.00	MEAN VAR Y	=	2.64
SD VAR X	=	142.829	SD VAR Y	=	0.329
CORRELATION	=	-0.995			
REG COEFF	=	0.002	REG CONST	=	3.156
SE OF COEFF	=	0.000	SE OF CONST	=	0.016
T-VALUE	=	-36.956	T-VALUE	=	192.655

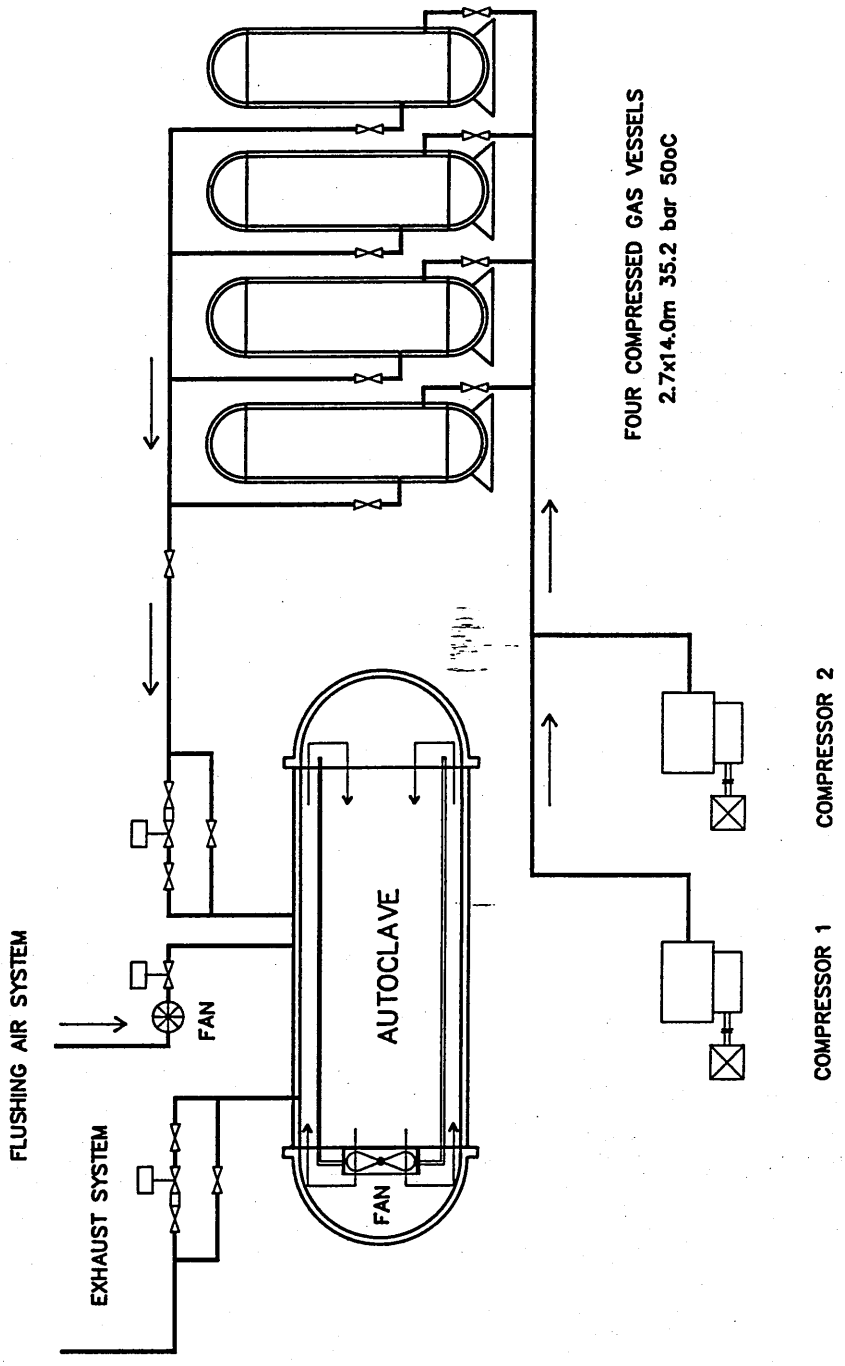
REGRESSION TABLE

SOURCE	SUM OF SQUARES	DF	MEAN SQUARE	F-VALUE
DUE TO REG	1.61	1.	1.61	1365.77
ABOUT REG	0.02	14.	0.00	
TOTAL	1.62	15.		

Regression Analysis of Results from Experiments on a Model Autoclave (Fan OFF)

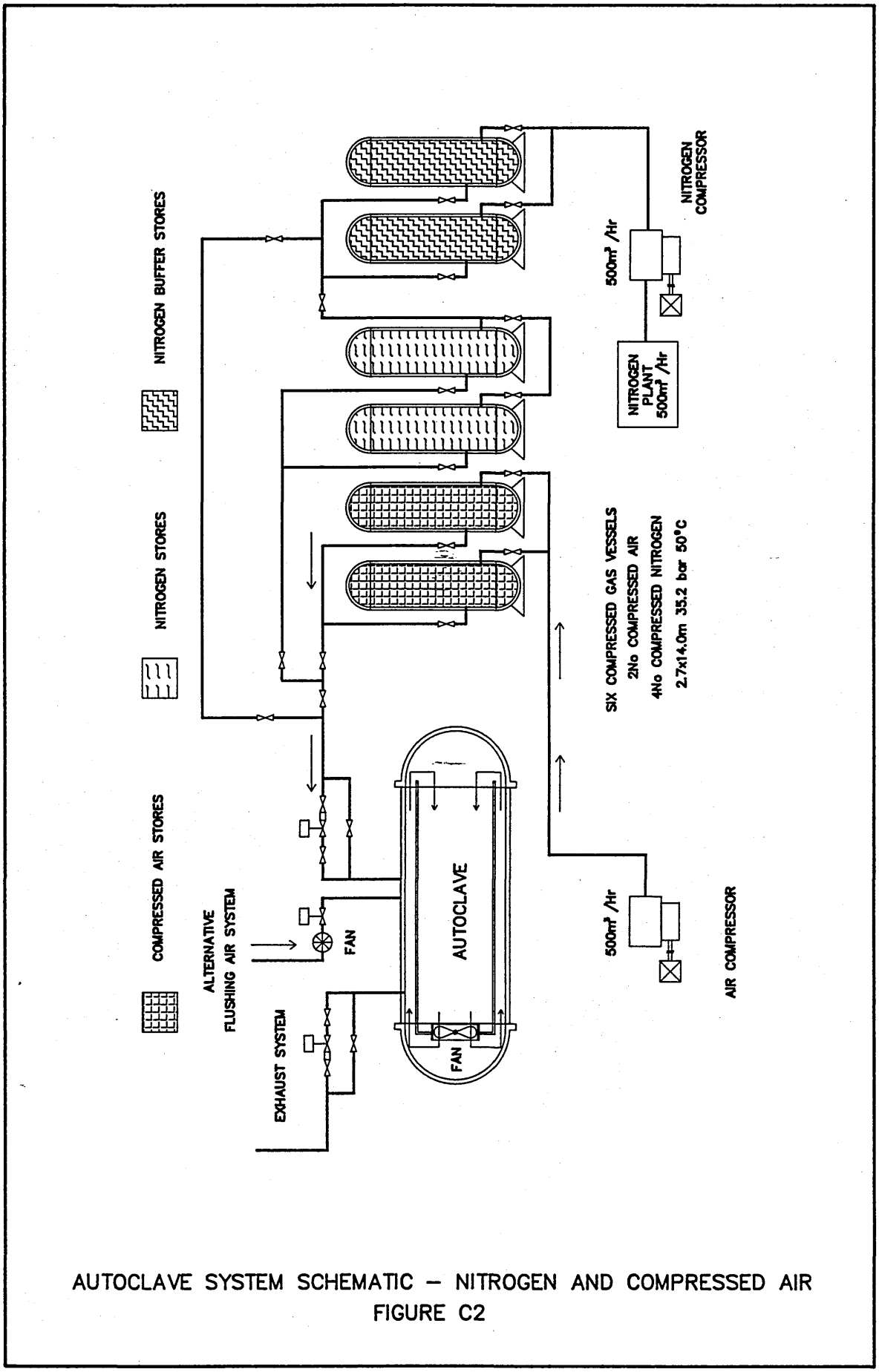
TABLE B.4

APPENDIX C



AUTOCLAVE SYSTEM SCHEMATIC – COMPRESSED AIR

FIGURE C.1



AUTOCLAVE SYSTEM SCHEMATIC – NITROGEN AND COMPRESSED AIR
FIGURE C2

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