OXYGEN FROM AIR BY PRESSURE SWING ADSORPTION

BY

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DECLARATION

The contents of this thesis represent my own work and the opinions contained herein are my own and not necessarily those of the Technikon.

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SYNOPSIS

The main objective of the work outlined in this project is to create an awareness of Pressure Swing Adsorption (PSA) Processes, their application to oxygen production systems in the RSA and the construction and testing of a pilot plant, built to assess an overseas design.

Available oxygen PSA technology was examined from a theoretical approach, right through to practical applications. The aim was not to re-invent the technology, but to review what technology is available and to assess its suitability for the South African Industrial Sectors. This was undertaken by investigating all PSA processes that are available to market the product to industry.

The technology review includes an investigation of overseas PSA technologies and compares these modes of technology with the technology that is readily available to Afrox Limited, a major gas supplier in South Africa. This resulted in the technology from the British Oxygen Company being thoroughly reviewed, examined and compared to various other modes of technology.

The basic principals of adsorption are discussed to give the reader an understanding of the factors that influence efficient adsorption and adsorbent regeneration. The parameters that define when adsorption separation processes are applicable to the separation of atmospheric gases are also discussed. The different types of PSA plant layout are discussed in great detail and it is explained when each plant layout type would be used. The focus is based on the economic viability of different types of PSA plant layouts available and their economical comparisons to the traditional methods of oxygen supply. The mechanical equipment required to achieve specific process conditions for the various PSA plant layout types are defined and put into practice during the operation of the pilot plant.

The construction, performance testing and results of the pilot plant show that PSA plants can be locally fabricated to high standards and can meet the output demands placed upon them. The pilot plant was commissioned with minimum effort and achieved a stable operating mode in a short period of time. The effects of ambient changes due to high altitude and atmospheric temperatures were not significant nor were they detrimental to the separation process in any way. The pilot plant achieved all of its design parameters with respect to

product flow and purity.

This project has resulted in the successful completion of the first operational oxygen PSA plant in the Republic of South Africa. The study has indicated that when the market needs arise for this type of plant to be installed the technology is locally available and the plant can be designed and fabricated. The viability study shows that the cost of oxygen produced from PSA could have major cost saving benefits which would help to reduce manufacturing costs in certain industrial sectors.

The final analysis is that PSA plants will play a vital role in the South African gas production industry.

OPSOMMING

Die doel van hierdie verslag is 'n bewusmaking van die prosesse van Druk Verandering Adsorpsie (PSA) en die toepassing daarvan op suurstofproduksiestelsels in die Republiek van Suid-Afrika as ook die konstruksie en toetsing van 'n loodsaanleg om 'n buitelandse ontwerp te ontdeel.

Beskikbare PSA tegnologie is vanuit 'n teoretiese oogpunt benader en dan prakties toegepas. Literatuur ten opsigte van beskikbare tegnologie is bestudeer en die toepaslikheid daarvan vir die Suid Afrikaanse Industriële sektore is ondersoek. Dit is gedoen deur al die PSA-prosesse, wat beskikbaar is om die produk aan die industrie te bemark, te ondersoek.

Hierdie oorsig sluit 'n ondersoek van oorsese PSA tegnologie in en vergelyk dit met die tegnologie wat geredelik aan Afrox Beperk, 'n gasverskaffer in Suid-Afrika, beskikbaar is. Die tegnologie van British Oxygen Company is deeglik ondersoek en vergelyk met verskillende ander tegnologieë in hierdie veld.

Die basiese beginsels van adsorpsie word bespreek om vir die gebruiker 'n perspektief te gee van die faktore wat effektiewe adsorpsie beïnvloed asook die parameters wat bepaal wanneer adsorpsieskeidingsprosesse van toepassing is. Die onderskeie PSA-aanlegte word breedvoerig bespreek en dit word verduidelik wanneer elkeen gebruik sal word. Die verslag fokus op die ekonomiese lewensvatbaarheid van verskillende tipes bestaande PSA-aanlegte en die meganiese toerusting benodig om spesifieke prosestoestande te bereik.

Die konstruksie, werkverrigting en resultate van die loodsaanleg het bewys dat PSA-aanlegte, wat aan hoë standaarde voldoen, plaaslik opgerig kan word en sal voldoen aan die uitsetvereistes wat daaraan gestel word. Stabiele operasionele kondisies is binne 'n kort tydperk bereik. Die invloed van omgewingsveranderinge, as gevolg van hoogte bo seevlak en lugtemperatuur, het nie die skeidingsprosesse beïnvloed nie. Al die verwagte produksiesvloei-en suiweringsparameters is bereik.

Hierdie projek het die vestiging van die eerste operasionele PSA-aanleg in die RSA tot gevolg gehad. 'n PSA-aanlegte kan ontwerp en opgerig word indien die markbehoeftes dit vereis. Daar word ook getoorn dat dit geweldige ekonomiese voordele kan inhou en

produksiekoste baie kan verminder vir sekere industriële sektore.

PSA-aanlegte sal dus in die toekoms 'n uiters belangerike rol in die gasproduserende industrieë van Suid Afrika speel.

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NOMENCLATURE

Symbol	Title	Units
A	Pore Area	$Å^2$
B	Langmuir dispersion constant	J
C	Dispersion constant	J
C_p	Specific heat at constant pressure	J/Kg.K
C_v	Specific heat at constant volume	J/Kg.K
E	Potential energy	J
F	Field strength	J
G	Gibbs free energy	J/Kg
H	Enthalpy	J/Kg.K
P	Total pressure	Pa
P_s	Vapour pressure	kPa
P_g	Saturation pressure	kPa
P_o	Vapour pressure on plane surface	Pa
Pwr	Power	kW
Q	Heat of adsorption	J
R	Gas constant	J/Kg.K
RH	Relative humidity	%
S	Entropy	J/Kg.K
SpPwr	Specific Power	$kWhr/m_n^3$
T	Temperature	K
V_N	Volumetric flow rate	m_n^3
V_s	Swept volume	m^3
b	boltzmans constant	J
d	partial derivative	-
e	natural log function	-
f	partial function	-
m	molar mass of adsorbate	mol
n	total amount adsorbed, specific heat ratio	mg,-
r	pore radius	Å
r_k	radius of curvature	Å

v	volumetric amount adsorbed	m^3
z	displacement	Å

GREEK

α	Separation factor	-
ß	Desorption constant	J
Θ	Contact Angle	Radian
μ	Chemical potential	J/mol
π	Spreading pressure	$J/Å^2$
p	Product recovery	%
σ	Interfacial tension	J
ϕ	Potential energy function	J

SYMBOLS

AlO_2	Aluminium Oxide
Ar	Argon
Ca	Calcium
Hg	Mercury
H_2O	Water
N_2	Nitrogen
Na	Sodium
M	Manganese
O_2	Oxygen
Si	Silicon
SiO_2	Silicon Oxide

Note, - implies dimensionless units

1 INTRODUCTION

Oxygen from air by pressure swing adsorption is set to revolutionise the South African industrial sector. It not only offers to reduce costs of most industrial oxygen applications, but it will also open paths to many new applications that were not viable due to the high costs of oxygen produced using cryogenic methods.

This statement is proving itself to industry. In several first world industrial countries, such as Japan and America, industries are marketing PSA systems on a nationwide basis. PSA systems can produce oxygen gas at a wide range of pressures, purities, flow rates and temperatures.

This thesis has been chosen as a result of the author having been given the task to study, through an international framework and available literature, all aspects pertaining to PSA technology and to establish this technology in the Republic of South Africa. The information compiled is used to guide the gas production industry in terms of alternative process cycles, the mode of technology, the type of plant, the effects of capital costs and the power requirements that would best suit South African industry.

Data and information has been sourced through colleagues in various operating centres, including the United States of America, the United Kingdom and Australia. The author travelled to Japan to source relevant information on this subject. This proved to be difficult due to the language barrier and cultural differences involved with the nature of business transactions undertaken in Japan.

The main objectives of this study are listed below :

a) The evaluation of the various PSA plant layouts, a review of process plant manufacturers and a brief comparison of these to the British Oxygen Company.

b) The understanding of the basic chemical process of adsorption technology;

c) The application of existing oxygen PSA technology and its economic viability to the South African industry;

d) The building of a pilot plant as a technology demonstrator to enhance local understanding of PSA technology and to verify an overseas plant process design package.

The results of the pilot plant mentioned in this text will be used to develop a generic PSA plant design package to be used by the British Oxygen Company (BOC) head offices in the United Kingdom. This will enhance the strategic position of the BOC group to be able to supply ultra modern PSA plants to the international industrial market.

Development of PSA technology is progressing continuously all over the world. Airco (USA) is currently commissioning a 30 tonne per day (TPD) oxygen PSA plant (February 1993) to supplement existing plant capacity and as a learning exercise. Commonwealth Industrial Gases in Australia are planning a similar exercise on a 22 TPD PSA unit.

After cryogenics, (producing oxygen at very low temperatures, typically -186°C (87K)), the most widely used process for air separation is adsorption (Thorogood, 1991). This is mainly due to the traditional methods of oxygen production being utilising cryogenic methods. Table 1.1 outlines the main difference in process plant performance for oxygen production by cryogenic and adsorption methods. The figures in this table have been compiled from provisional BOC PSA plant designs, and from existing cryogenic plants being operated by Afrox Limited in June 1992.

PSA generated oxygen in the total scenario is more limited in scope than cryogenic oxygen. Cryogenic oxygen is transportable at a 20 ton maximum load and therefore a great volume can be stored on site and vaporized into a gas when required. Cryogenic oxygen also features a typical purity of 99,7%, the balance being a noble gas, argon. PSA oxygen cannot economically meet these high purity criterion and will therefore only be suitable to replace approximately 30% of the current South African oxygen applications.

Item	Unit	Cryogenics	Adsorption
Typical Purity	% O_2	99,7	93
Minimum Flow	m_n^3/hr	600	0,1
Other products		N_2/Ar;	None
Pressure	Bar(g)	0,4	Variable
Unit Costs	R/Tonne	78	117
Specific power	kWhr/m_n^3	0,39 - 1,7	0,45 - 0,9
Maint. Requirement	R/Tonne	30	15

TABLE 1.1

CRYOGENICS vs ADSORPTION

Replacing 30% of the cryogenic oxygen applications may leave the South African gas production industry in quite a predicament. The capital and power intensive gas liquefiers, termed Air Separation Units (ASU), typically liquifying nitrogen and then by direct contact liquifying air and distilling out the oxygen, would be left under-utilised. The result is generally that a long term payback on capital investment cannot be met.

The information outlined in this thesis is a collection of relevant data regarding PSA technology. All recommendations and information are built around new applications, which would be financially viable. Emphasis is placed on the mechanical operating characteristics and commercial implications (operating and capital costs) of different types of PSA plants.

It is of great importance to note that this thesis focuses on a chemical engineering process. Therefore, many units of measurement are specified in process engineering terms so as to facilitate a complete measurement of a mass balance at all times. These terms relate mostly to flow measurement being specified at certain reference conditions which are internationally used in chemical engineering process design.

2 LITERATURE SURVEY

2.1 GENERAL

A literature survey was undertaken through the British Oxygen Company (BOC) international network. It was discovered that very little has been written on PSA applications to separate atmospheric gases. Books were recommended by European and American sources and these were obtained locally.

Mintek library, CSIR library and various university libraries were visited. The keywords used were "Separation of Gases". The most effective manner of keeping abreast with the technology was to monitor several gas separation journals and periodicals. Subscriptions were arranged for various journals as listed in the reference section of this thesis.

It was also found that most chemical engineering texts contained information regarding adsorption separation processes, which were usually aimed at applications in the petro-chemical industry (Perry, 1988 : 16-24) and these made good informative reading.

2.2 PSA TECHNOLOGY

There are various modes of PSA technology appearing globally and it is of utmost importance to clarify the relationship between these plant layouts. Pressure Swing Adsorption or PSA is the generic term given to the type of plant that is used to separate a mixture of gases using adsorption principals and incorporating a change of pressure on the molecular sieve during the process. PSA plants have been used in many cases in the past to separate hydrogen from the light flue gases in the petrochemical industry. Only recently have PSA technologies been applied to the separation of oxygen from the atmosphere.

The various types of PSA plant layouts that are suitable for the separation of atmospheric gases are known as PSA (Pressure Swing Adsorption) plants and VSA (Vacuum Swing Adsorption) plants. A VSA plant will operate at lower pressures than a PSA plant and it incorporates a vacuum pump to regenerate the zeolite molecular sieves that are necessary for the separation process. These two plant layouts are discussed in greater detail in the following sections.

2.2.1 PSA System Performance

It is necessary to split the available PSA plant types into three groups, depending on the plant output capacity. This is essential since these different plant types have affects on purity, pressure, proportional capital costs, installation time, process cycles, turndown capacity and compatibility to the system that is requiring the product oxygen gas. . All these factors have to be investigated and considered and they need to be tailored to best suit the specific requirements of the oxygen application.

2.2.1.1 Small Oxygen PSA Units

Most small oxygen PSA units could be used primarily in hospitals or in small engineering works. There could be a large demand for these PSA units in South Africa especially in remote areas where the delivery of high pressure oxygen gas cylinders is expensive. An example of a small oxygen PSA application is found in Phalaborwa, where a Ramfab PSA unit is being used for scrap metal cutting. This plant supplies sufficient oxygen gas to meet the requirements of two cutting torches.

Hospitals can be supplied with PSA oxygen if the gas produced meets an international specification for oxygen used for medical purposes, called the European Pharmacopoeia.

A PSA unit can produce oxygen to comply with this medical specification when it is fitted with special filters, valves, oxygen analyzers and instrumentation. This will of course increase the capital costs of these units. Table 2.1 shows the typical PSA oxygen purity obtained by a BOC Medflow 5000 medical oxygen PSA unit operating on a test in February 1992.

There are various units readily available which can be purchased from several suppliers, predominantly Japanese. The only known medical oxygen unit in South Africa is a test unit running at Baragwaneth Hospital in the Transvaal. This unit was supplied by Airsep, an American PSA manufacturing company. The BOC group has several units running in the Harley Street Clinic in London. These appear to be the only medical oxygen PSA units operating in the United Kingdom. The author will be installing a medical oxygen PSA unit into a private hospital for performance testing and evaluation in October 1993. This is a two bed pressure atmospheric PSA unit that has been imported from the United Kingdom and is shown in Figure 2.4. Medical oxygen PSA units are only expected to be installed in hospitals in remote areas where the delivery cost of cryogenic liquid oxygen is expensive. The medical oxygen PSA plants installed in various hospital applications overseas have not been well received due to problems associated with noise levels and the maintenance requirements that are associated with PSA plants are very different to the normal hospital maintenance systems. These problems will have to be addressed before the introduction of medical oxygen PSA units into South African hospitals.

Constituent	Volumetric %
Oxygen	93
Argon	3,5
Nitrogen	3,5
Moisture	< 2 ppm
Hydrocarbons	< 1 ppm

TABLE 2.1

TYPICAL PSA OXYGEN PURITY (BOC MEDFLOW 5000, 1992)

All small oxygen PSA units will operate on the two bed pressure atmospheric cycle. This cycle requires a package type screw compressor to deliver air to the plant at a high pressure. With this high pressure air feed, the product can be supplied at a high pressure, thus eliminating the need for product oxygen compression. Vacuum equipment is not needed and with only two pressure vessels, capital costs are kept to a minimum.

Small oxygen PSA units could become the main business area of a small gas supplying business.

2.2.1.2 Intermediate Oxygen PSA units

Most intermediate size oxygen PSA units could be used in industrial applications, for furnace enrichment or gas cylinder filling in remote locations. These units are not readily available and have to be designed, manufactured and installed according to the specific requirements of the customer. The Japanese appear to be the market leaders for this type of PSA unit, closely followed by an American company called Airsep. There are no known units of this size operating in South Africa.

Intermediate size PSA units could operate on various process cycles. The most likely is the low capital cost two bed pressure atmospheric cycle. This two bed cycle, explained in section 2.3.1 uses approximately 50 percent more power than the three bed cycle, explained in section 2.3.2. However, if the oxygen application should require a plant with a low operating power demand, the three bed pressure vacuum cycle would be considered. The choice of process cycle would also be dependent on the required pressure, purity and flow of the product oxygen.

Initial estimates indicated that this range of oxygen PSA unit may not be economically viable in South Africa. However, the situation is rapidly changing due to new sieve types and process optimisation, resulting in better cycle efficiencies and therefore lower cost plants.

2.2.1.3 Large Oxygen PSA plants

Large oxygen PSA plants would be built on a customers site as a major oxygen supply scheme, typically to a mine or to the Iron and Steel Industry. These PSA plants would either be installed as a stand alone supply scheme, or to supplement an existing cryogenic oxygen air separation unit, if the customer could accept a slightly lower purity product.

All large oxygen PSA plants have to be designed to meet the customers specific requirements. The choice of process cycle is limited, as the two bed pressure atmospheric cycle reaches a maximum product output capacity of nearly 30 TPD. At this size, it is still debatable as to which cycle would be used. A supply greater than 30 TPD could also be achieved by using more than one train of two bed plants or a single train three bed plant.

The three bed cycle efficiency may sometimes be improved by adding four, five or sometimes even six beds to the PSA process. The Japanese appear to be technological leaders in this type of efficiency modification.

All large oxygen PSA plants appear to compare favourably against the costs of cryogenic oxygen process plants up to approximately 100 TPD (Labrousse, 1989). It is expected that an oxygen supply above 100 TPD would be more viable if supplied by a cryogenic oxygen process plant.

A case study completed by the author in November 1992 for an oxygen supply of 130 TPD to Rustenberg Platinum Mine (RPM) indicated that a PSA oxygen plant was approximately 30 percent more costly than a low purity cryogenic oxygen generator.

The operating characteristics of different sized BOC plants are shown in Table 2.2, (BOC Novox PSA plants, 1992).

Item	Unit	Small	Intermediate	Large
Capacity	TPD	0 - 5	5 - 20	20 - 100
Air Supply	Bar(abs)	8	8	1,4
Product Pressure	Bar(abs)	5	2	1,2
Typical Purity	% O_2	80 - 90	90 - 93	93
Specific Power	kWhr/m_n^3	0,7 - 1,1	0,4 - 0,9	0,45
No. of Beds		1 - 2	2 - 3	3 - 4

TABLE 2.2

SMALL, INTERMEDIATE AND LARGE OXYGEN PSA UNITS.

2.2.2 Installations

The BOC Oxygen PSA Centre of Excellence conducted a worldwide survey of operating oxygen PSA plants in February 1992. This was done by contacting the main manufacturing companies and obtaining lists of operating plant installations. The results are shown in Figures 2.1, 2.2 and 2.3 for total PSA oxygen capacity, the number of plants installed and their worldwide distribution (BOC Centre of Excellence annual report, 1992).

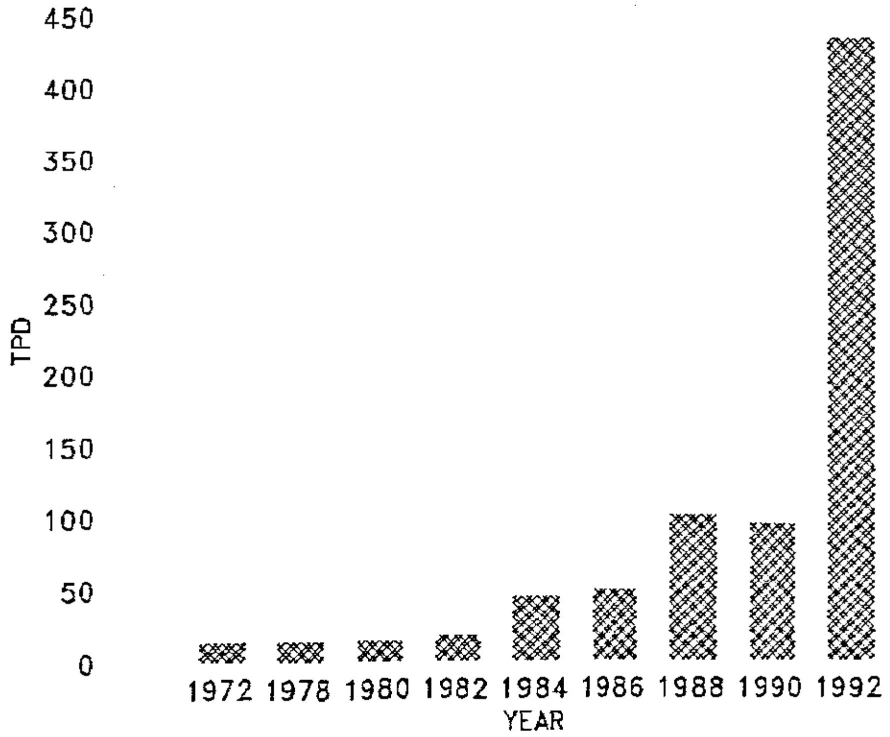


FIG. 2.1

WORLDWIDE OXYGEN PSA CAPACITY

Figure 2.1 shows that the total installed PSA plant capacity per year is steadily increasing, (the plant figures for 1992 are estimates, based on globally reported work in progress), while Figure 2.2 indicates that the number of large PSA plants installed per year is decreasing. This is most likely as a result of the current worldwide economic recession. This also implies that PSA plants are being manufactured to produce a larger volume of product oxygen. Figure 2.3 shows the worldwide PSA plant distribution, which indicates that most of these plants are located in Asia, with a small plant distribution in the other continents.

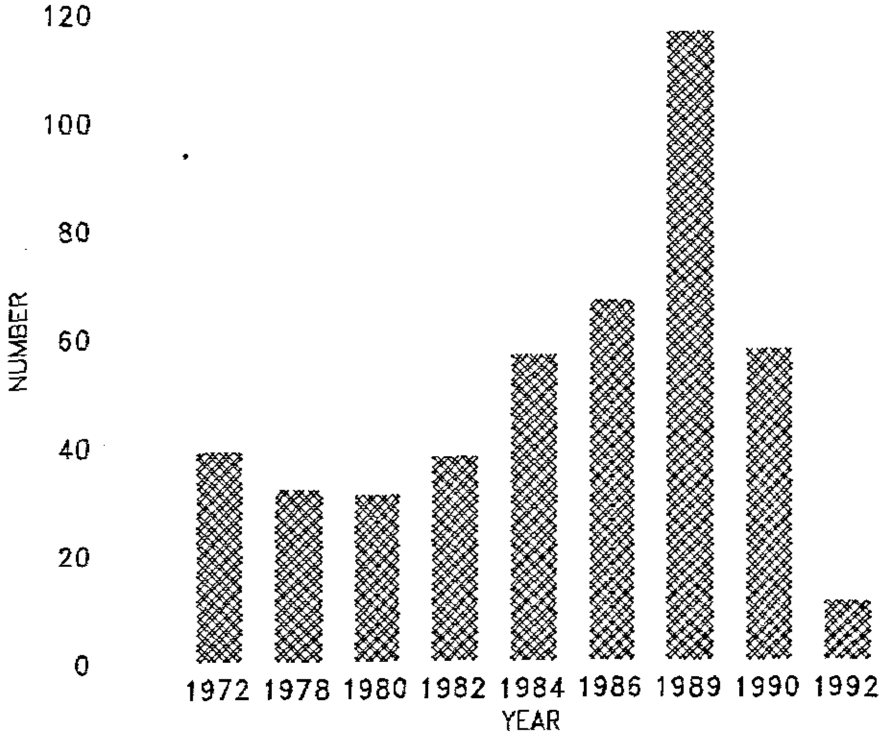


FIGURE 2.2

INSTALLED OXYGEN PSA UNITS

.2.3 Cycle efficiency

PSA plant cycle efficiency can be measured in terms of the percentage recovery of the available oxygen in the feed stream to that in the product stream. This measurement is simply termed "Product Recovery". The product recovery is dependent on the operating characteristics of the PSA plant and the type of molecular sieve used. Product recovery is in some cases referred to as yield.

PSA plant cycle efficiency can be more practically and mechanically measured by the plant specific power, a term used to indicate the plant power requirements in terms of kilowatt-hours per normal cubic meter of contained oxygen (i.e. $kWhr/m_n^3$ oxygen). This measurement is obtained by measuring the plant volumetric output flow rate and adjusting this figure to normal conditions at a pressure of 101,3 kPa (abs.) and at a temperature of 0°C by using Charle's gas law. This measurement is then corrected to a contained oxygen flow rate by dividing by the product purity. The kilowatt hour meter reading is measured for the mainline equipment only and this is recorded at the motor input terminals, which does not incorporate the cable and distribution losses through the electrical transmission.

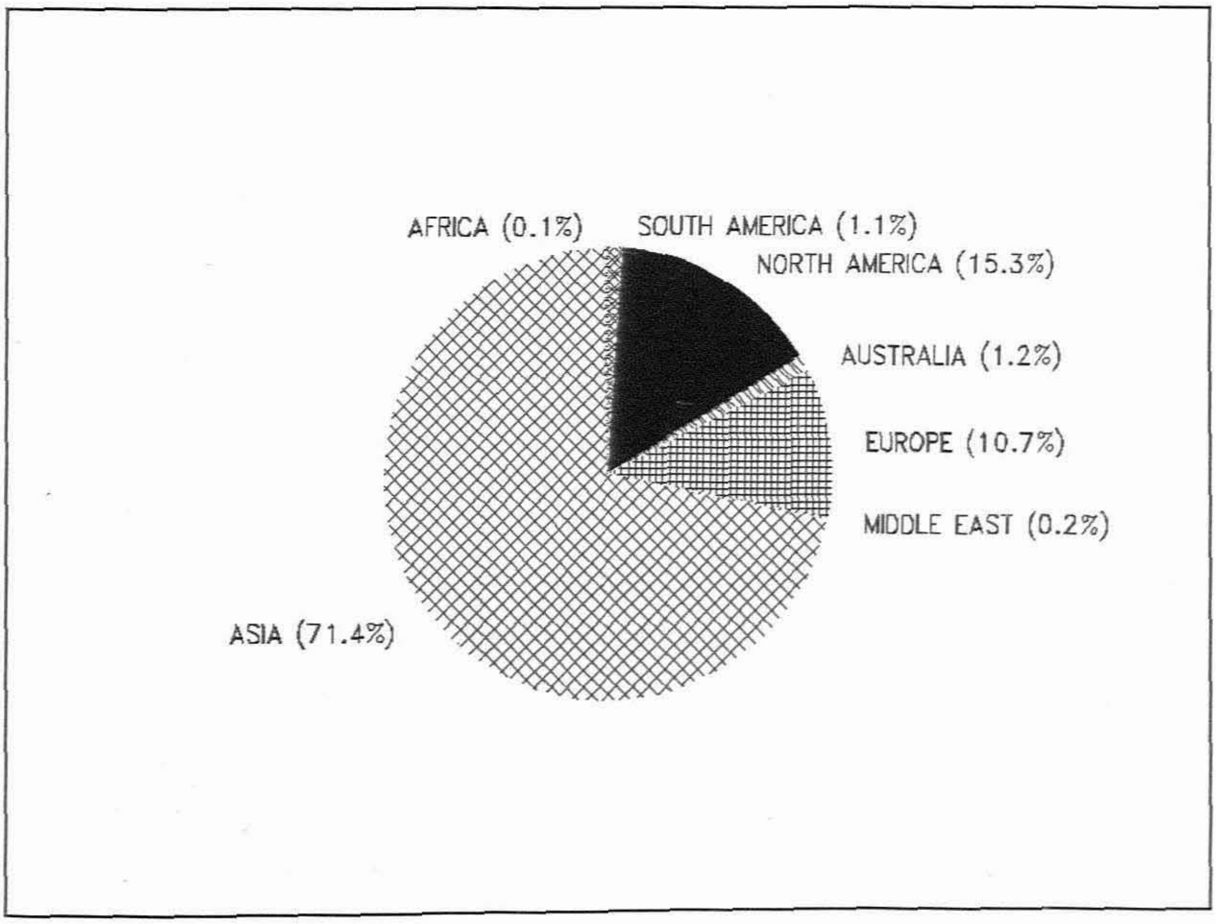


FIGURE 2.3

WORLDWIDE PSA PLANT DISTRIBUTION

The efficiency, or the usefulness, of the zeolite molecular sieve is represented by the specific product. The specific product is defined as the quantity of gas produced in terms of normal cubic meters per hour per cubic meter of molecular sieve installed in the plant. This molecular sieve characteristic also defines the amount of sieve that is required for the plant to operate.

2.2.4 PSA Oxygen Applications

PSA generated oxygen is more limited in scope than cryogenic oxygen due to the lower purities obtained. However, various oxygen applications are being operated on high purity oxygen because until recently, high purity oxygen was the only commercially available oxygen product. Oxygen applications that would be ideally suited to PSA oxygen are those with a continuous demand pattern. A continuous demand pattern implies that the application requires a constant oxygen flow rate for a steady period of time, usually not less than 24 hours, every day, seven days a week. These applications include : Fish farming, Gold leaching in cyanide, Furnace enrichment, Ozone generation, Waste water treatment, Glass manufacture and the Healthcare industry. It is also possible to use PSA oxygen for gas welding and flame cutting however, the fuel gas consumption will be higher, due to the decrease in oxygen purity.

2.3 PLANT LAYOUTS

As discussed briefly in Section 2.2, there are many different options available for oxygen PSA applications. These options are based on the theoretical considerations mentioned in Chapter 3. The two main types of PSA plants, namely pressure atmospheric plants and pressure vacuum plants, will be described in a practical manner, from which a conclusion and recommendation can be made regarding their suitability to the South African industrial sector.

2.3.1 Pressure atmospheric plants

Pressure atmospheric plants are currently being developed in most major first world centres. These plants are predominantly known as low capital and high power cost plants. They are usually used to supply a user with a low product demand, typically less than 10 TPD.

Pressure atmospheric plants are operated on a high pressure two bed process. This process involves a high pressure air feed to be admitted to the two PSA adsorber vessels. The typical plant operating pressure range is between 7,5 bar(g) and the relevant atmospheric pressure. The maximum product pressure is that of the feed air pressure and in order to achieve this a larger air flow is required but in most cases this is not economically viable due to the high costs of compressed air. The process operates between atmospheric pressure and the feed air pressure. The molecular sieve regeneration is achieved by venting the waste stream to the atmosphere, causing a lower pressure on the bed and thus allowing the nitrogen molecules to be desorbed from the molecular sieve, to return the molecular sieve back to its original unsaturated condition. A typical two bed oxygen PSA system is shown in Figure 2.4. This unit will produce up to 5 m_n^3/hr or 0,17 TPD. The unit is to be installed and an air compressor, refrigerant drier, air receiver vessel and product buffer vessel still need to added to this system.

FIGURE 2.4
PRESSURE ATMOSPHERIC PSA UNIT

2.3.1.1 Mainline Equipment

The high air feed pressure, typically 7,5 bar(g) is usually supplied by using an oil free screw type compressor. These units are readily available in a package form and incorporate a complex control philosophy (Atlas Copco). With the addition of an air receiver vessel a steady supply of process air is obtained. These air compressors are readily available from compressor manufacturers such as Atlas Copco and Aerzen. It will be necessary to add a refrigerant drier in certain cases, especially where the PSA plant is installed in a warm, humid environment.

The pressure vessels for the adsorbate need to be designed to incorporate cyclical loading conditions. The trend in design for these vessels complies with ASME VIII Div. 2 (BOC MEDFLOW 5000, 1992). The cyclical load typically varies between 7,5 bar(g) and atmospheric pressure. The product and air buffer vessels are designed according to the operating pressure only as they are not exposed to the cyclical pressure conditions of the adsorber vessels due to the addition of pressure control valves and non return valves.

The interconnecting pipework between the adsorber vessels is fairly simple. Copper pipe is usually used on the oxygen product side of the PSA plant to cater for the critical restrictions placed on high pressure oxygen gas pipeline velocity. A typical two bed pressure atmospheric plant requires eight process actuated valves. These valves are usually double acting pneumatically controlled high performance butterfly valves designed to perform over one million cycles per year operating at 24 hour a day.

The plant control and process instrumentation is achieved by using simple pre-programmed electronic timers and analyzers. The plant control panel typically absorbs about 200 Watts for all types of two bed oxygen PSA plant. The instrument air requirements are supplied from the main air compressor outlet, downstream from the oil and dust particle filters, where instrument quality air can be obtained.

The basic PSA unit is typically factory manufactured and delivered to site in one package for plants up to about 5 TPD. The total installation time is usually 15 working days which includes the plant commissioning. Factory lead time is typically six months. Larger plants are built on site with installation time ranging between six and eighteen months.

2.3.1.2 Cycle Efficiency

As outlined above, this cycle uses a minimum requirement of mainline equipment. Logically, a two bed PSA plant layout produces a lower product recovery than a three bed cycle as the useful oxygen product cannot be re-circulated efficiently between two beds. The typical product recovery of a two bed PSA plant is usually between 30 and 40 percent depending on the plant operating characteristics and the choice of molecular sieve. Air feed at a pressure of 7,5 bar(g) requires a high compression power. Due to these factors, a low product recovery with high specific power is achieved when using this two bed cycle. The typical plant specific power is 0,7 to 0,9 kWhr/m_n^3 contained oxygen. (Airsep, 1991).

2.3.1.3 Plant Outputs

Pressure atmospheric PSA units typically produce oxygen at 90% product purity. By increasing the cycle time and the bed height, purities of 93% to 95% can be obtained. This will always significantly increase the plant specific power and will require a larger air compressor to deliver the greater quantities of air that are required to meet higher production purities and pressures. The economical product output pressure ranges between 1 Bar(g) and 4 Bar(g), depending on the air feed pressure and the mode of plant operation.

This process cycle has been successfully applied to a complete range of units, with capacities from a few litres per minute to tonnage scale plants. The largest two bed pressure atmospheric plant installed to date is in New Zealand, producing 34 TPD (Airsep, 1991). At the other end of the scale, this process cycle also provides oxygen for home and for medical uses.

This process is very easy to scale up or down. In order to double the plant production, all that is required is to double the airflow, double the mass of adsorbate and to calculate pipe diameters to give a constant gas velocity in the process pipework. The total output from the plant can be designed and built to achieve specific oxygen supply requirements.

2.3.1.4 Manufacturers

There are various manufacturers of two bed pressure atmospheric PSA units. The predominant suppliers are Air Separation Technologies, known as Airsep, various Japanese suppliers and the British Oxygen Company.

Air Separation Technologies have the largest customer reference list and as a result appear to be world leaders by comparison of experience and installed plant capacity.

Many Japanese suppliers produce two bed oxygen PSA systems. However, due to the economical situations in Japan, it is far more economical for the Japanese to build three bed oxygen VSA systems.

The British Oxygen Company have designed a range of two bed plants and to date have only two reference plants in operation. Two further plants are expected to be commissioned during the course of 1993 in Nigeria and India producing 1 TPD and 8,5 TPD respectively.

In South Africa it is not expected that these plants would be locally manufactured. It is most likely that these would be imported as a unit and installed and commissioned by local engineering personnel.

2.3.2 Pressure Vacuum Plants

Pressure vacuum, or VSA plants, are currently being developed by numerous process plant manufacturers. Many of these plants are operating in Japan, where suppliers appear to be world leaders in this technology. These plants are predominantly high capital cost units and have a very low absorbed power requirement. They are used to supply the upper product capacity range with plant output design capacity greatly exceeding the capacity obtainable by the two bed pressure atmospheric plant.

Pressure Vacuum Plants are operated on a low pressure vacuum and atmospheric pressure cycle. Typical pressure ranges for this type of VSA is between 200 mbar (abs.) and 1500 mbar (abs.), depending on the molecular sieve characteristics. The production and adsorption steps are performed at the pressure of 1500 mbar (abs.), which is slightly above coastal atmospheric pressure. The molecular sieve is regenerated by drawing a vacuum on the bed, causing a desorption of the adsorbed gas, nitrogen. Vacuum swing adsorption plants generally absorb less power than the pressure swing-type, due to the lower operating pressures and the higher operating efficiencies obtained by using more than two adsorber vessels.

2.3.2.1 Mainline Equipment

The mainline equipment outlined in this section forms the basis of the hardware involved in the construction of three bed oxygen VSA plants. The choice of machinery is discussed in greater detail in section 3.3.2, which relates to the design of a three bed VSA plant built as a pilot plant and as a technology demonstrator in South Africa.

The low feed air pressure requirement is approximately 1500 mbar (abs.) and is obtained by using a Roots or centrifugal type blower. This must be an oil free unit, as oil is a catalytic poison on the molecular sieve and any contact with oil will severely degrade the adsorption capacity of the molecular sieve. The vacuum pump is usually of the water injected Roots type, with two stages and approximately twice the volumetric capacity of the pressure blower. The vacuum cycle at its lowest point usually reaches approximately 200 mbar (abs.). The Roots type pressure and vacuum equipment presents specific problems associated with high noise and vibration levels. Acoustic canopies and rubber anti-vibration mountings are usually supplied and installed to limit these shortcomings.

The pressure vessels to contain the zeolite molecular sieves are designed to accommodate for cyclical loading conditions. The usual design code is ASME VIII Div. 2 (BOC, V1000, 1988) or similar accepted design codes. The cyclical load varies according to the process design criteria, usually between 200 mbar (abs.) and 1500 mbar (abs.) every minute. The product buffer vessel is also built to this design code, as in the case of a valve failure, it may be possible to expose the product buffer vessel to the full vacuum condition. The vessels are usually manufactured by using a minimum thickness of 20 mm steel plate. The vessel shell thickness will increase as the vessel diameter increases and at times the vessel design may incorporate vacuum stiffening rings to be attached to the outside of the vessel shell. The air pressure and flow rate into the adsorber vessels is controlled by an automatic pressure controller and pressure control valve so that there is no requirement for a air receiver vessel on the air blower discharge pipework.

The interconnecting pipework between the vessels is fairly complex due to the purge, backfill and top to bottom equalisation part cycles. A typical three bed pressure vacuum plant will require eighteen automatically operated process control valves to achieve these part cycles. These valves are generally double acting pneumatically controlled high performance butterfly valves, performing on average, one million cycles per year, when operating 24 hours a day. These valves can range from 150 mm to 600 mm in diameter, depending on the plant output capacity.

The plant control and data recording instrumentation is achieved by using complex pre-programmed Programmable Logic Controllers (PLC) and process flow analyzers. These usually require a large degree of adjustment and fine tuning during the plant commissioning phases. The plant control panel typically adsorbs about 400 Watts for all types of VSA plants. Instrument air is supplied from a separate instrument air compressor package or a high pressure gas cylinder manifold system as the air to plant feed pressure is too low to be successfully used for instrumentation purposes.

These VSA plants are usually built on site, due to the size of the plant which is not easily transportable. The total installation time ranges from nine to eighteen months, depending on the plant output capacity and the major equipment lead time.

The product to the customer is usually supplied under pressure by using an oil free labyrinth type reciprocating compressor. These can only be obtained from a few specialised manufacturers with a long delivery time and are expensive. Alternative oxygen compression systems include a liquid ring machine that may be used in an application such as waste water treatment or fish farming where the dryness of the oxygen gas is not an important criteria.

2.3.2.2 Cycle Efficiency

Due to the nature of the three bed cycle, a portion of the product oxygen is used to backfill, purge and to pressurise the other beds and this assists with the molecular sieve regeneration process. This cycle produces a product recovery between 25 and 50 percent, depending on the operating cycle characteristics and the choice of molecular sieve. The feed air is compressed to a low pressure, requiring a low compression power. Vacuum equipment can be selected to limit power consumption. Due to all of these factors a very low plant specific power is achieved on the three bed cycle. A typical specific power of 0,4 to 0.45 $kWhr/m_n^3$ contained oxygen with a product recovery of 25 percent is obtained by Showa three bed VSA plants in 1992. This power figure can be reduced even further by using 4,5 or 6 beds which will also increase the product recovery.

2.3.2.3 Plant Outputs

Pressure vacuum plants typically produce product oxygen at 93% purity. Product purity can be increased to 95% for a substantial power penalty. This increase in purity will have a corresponding decrease in the product take off rate. Product gas pressure is approximately 1200 mbar (abs.) and would require further compression, if a higher product supply pressure is required.

This process cycle has been successfully applied to plants ranging from 1 TPD up to 130 TPD. The largest feasible plant design as indicated by the Japanese to date is a 210 TPD plant (Showa, 1991). It is not economical to build these plants to produce below 5 TPD due to the high capital cost of plant construction.

The three bed vacuum cycle can easily be scaled between various sizes up to about 90 TPD. The critical factor is the molecular sieve bed height and for doubling the production, it is necessary to double the cross-sectional area of the bed and to maintain a constant bed height. This keeps the power requirements to a minimum as the gas flow pressure drop over the molecular sieve bed is not increased. Pipe flow velocities must also be kept constant. The total output from these plants can be designed and built to achieve specific customer requirements.

Scaling up process designs of the three bed VSA cycle leads to several shortcomings. The author received Showa VSA process design outlines during a visit to Japan for 130 TPD and 165 TPD plants. The construction of these plants proved to be uneconomical mainly due to the large process pipe diameters required. The plant required pipework ranging from 900 mm to 1200 mm diameter pipes. Pipes of this size are not readily available and would have to be spiral wound from a 12 mm flat steel plate at a large expense. In addition to the pipework, the sheer volume of molecular sieve to be handled would be very labour intensive and the plant construction would require special cranes, drum hoisters and rigging gear which greatly increases the plant installation costs.

2.3.2.4 Manufacturers

There are numerous manufacturers of the three bed pressure vacuum VSA plants. The predominant market suppliers are the Japanese, where there are more than five process plant manufacturing companies. They have the largest number of reference plants in the world and are the predominant technological leaders in this market at present. Several European and American companies have indicated technological advances in this direction.

The BOC group have also developed this process and have a computer based plant design package available. To date, they have built two 30 TPD plants operating on these cycles. The pilot plant mentioned in this text is operating on the pressure vacuum cycle and it is built to a provisional 10 TPD BOC design package. This design will be verified during the pilot plant construction and performance testing.

2.3.3 Japanese Technology

Japanese Engineering Consultants Exports (JECE), an agent who deals between the RSA and Japan, was contacted regarding oxygen PSA systems. The main objectives were to evaluate Japanese technology, to identify who would do business in South Africa and to establish which manufacturer would be able to offer competitive PSA systems in South Africa. Japanese high pressure gas regulations require that cryogenic oxygen plants are constantly manned and as this requirement is not applicable to PSA plants, it reduces the operating costs of PSA plants and makes them more favourable than cryogenic plants. Numerous Japanese companies were contacted and four emerged with competitive technology and costs. These companies are Showa Engineering, Sumitomo Seika, Kofloc and Nippon steel.

2.3.3.1 Mainline Equipment

All Japanese plants examined were complete units supplied with Japanese mainline equipment. Low pressure air blowers, vacuum equipment, motors, valves and instrumentation were all from suppliers unknown to us in South Africa. Japanese suppliers were not prepared to guarantee their plant performance figures when utilising machinery that is not sourced by the vending company. This totally eliminated local mainline machinery and equipment supply.

The pressure vessels were all designed to accommodate for the maximum operating pressure ranges and the design did not allow for any degree of cyclical loading. The vessels were all built with external "H" shaped stiffening rings to prevent the vessels from collapsing under the vacuum conditions. The design code was Japanese Industrial Standards (JIS) (Showa, 1991). The JIS code will only be acceptable in South Africa if a full design review is undertaken by an inspection authority that is approved by the department of manpower and the Japanese manufacturing drawings have been approved before construction begins. The product and air buffer vessels are not specialised items and could therefore be designed and manufactured locally to acceptable design codes.

The interconnecting pipework varied between plant manufacturer and depended largely upon the nature of the process part cycles. This pipework was also predominantly designed to JIS. Valves were all high performance butterfly type, with double acting pneumatic actuators. Typical valve suppliers are Tomoe and NBS (Showa, 1991).

For capacities below 2 TPD, standard plants are factory manufactured in approximately one month. These plants are then installed on site in two days, including commissioning. Plants above 2 TPD are all custom designed and built on site. A typical plant lead time for installation is between 8 and 16 months, depending on the plant capacity. The longest lead time items are usually the molecular sieves and the product oxygen compressors.

2.3.3.2 Cycle Efficiency

Due to Japan having almost the most expensive electricity in the world (Japan imports all of its oil and coal), the emphasis is placed on operating power costs. Inflation in Japan is very much lower than that in South Africa which makes the cost of borrowed capital relatively cheap and the cost emphasis of PSA plants is placed on the operating costs and cycle efficiencies and not the plant capital costs. Due to these reasons, Japanese manufacturers are using the fan to vacuum cycle, usually with 3 or 4 beds. This fan to vacuum cycle operates with a very low air feed pressure supplied by a turbo fan and as the compression ratio in a machine of this nature is low, it is not necessary to aftercool the feed air. The vacuum levels achieved on the molecular sieves is very similar to that of the standard pressure vacuum cycle. The typical efficiency of a pressure vacuum cycle was discussed in section 2.3.2.2 and a fan to vacuum cycle has similar plant efficiencies. The Japanese plants tend to have far lower product recoveries than European plants, but the power consumption is usually less. The result of the low product recovery usually implies that the molecular sieve specific product is low which causes plant costs to increase due to larger vessels being required to contain the additional molecular sieve.

2.3.3.3 Plant Outputs

Japanese PSA plant manufacturers all normally construct plants to produce a product containing 93% oxygen. Any alterations to this is done as a deviation from a standard plant design. Purities ranging from 90% to 95% can however still be obtained at a substantially higher capital cost and power requirement.

Japanese manufacturers produce a large range of PSA plants. Their outputs vary depending on the process cycle that is being applied. The Japanese can supply plants to produce anything from a few litres per hour to 140 tonnes per day. Due to the very high electricity costs, most small plants (up to about 5 TPD) use the two bed PSA system. These plants include oxygen concentrators (sometimes single bed units, using a process of paramagnetic pumping. This is a Temperature Swing Adsorption (TSA) process that cannot be applied to larger units.) and small PSA units for medical and home uses. Japanese industries are currently manufacturing the worlds largest PSA unit to date, to produce approximately 210 TPD.

Product oxygen can be compressed from a low supply pressure to a high product pressure when required. Additional product oxygen compression is usually included in the package deal when purchasing plant and equipment from Japan.

2.3.3.4 Manufacturers

Four distinct manufacturers for PSA systems were identified. These were

Kofloc, Nippon Steel, Showa Engineering and Sumitomo Seika. Other familiar Japanese PSA manufactures included Kurrary Chemical Company, Nippon Sanso Corporation, Sanyo Electronic Industries, Mitsubishi Heavy Industries and Hitachi Limited. Mitsubishi is a large engineering concern who have complicated plant designs for a four bed VSA plant (Mitsubishi, 1992). Taiyo Sanso are still developing their oxygen PSA systems and are currently manufacturing nitrogen PSA systems (Taiyo Sanso, 1990).

Kofloc is a long established company dealing mainly with small PSA units, up to a capacity of 0,25 TPD (Kofloc). These are obtainable within one month from the receipt of an order, manufactured, packed and ready for export. Costs for these units were obtained from JECE for evaluation and these plant were found to be expensive.

Nippon Steel Corporation (NSC), is the largest steel company in the world. Nippon Steel have successfully completed work in South Africa by manufacturing and installing the iron and steel mill used in the ISCOR works at Vanderbijl Park. They have their own in-house developed oxygen PSA technology and use their own PSA plants at their steel mills (Nippon, 1990). There is no evidence of them having sold a PSA unit to a third party and it is not practical for them to export PSA plant and equipment. They use a readily available Japanese manufactured molecular sieve and their future commitment to oxygen PSA production is unknown.

Showa Engineering Company (SEC) is an engineering division of Showa Denko, who employ 450 engineers. Showa Engineering has successfully completed work in South Africa by installing the Consolidated Metals Industries (CMI) plant in the Transvaal. Showa Engineering have developed their own in-house PSA technology and claim to be the largest PSA plant supplier in the world in terms of installed plant capacity. They have exported various units to several first world countries, including Ireland, and appear to be willing to do business in South Africa. They use their own zeolite molecular sieve called Union Showa sieve. They appear to be a world class supplier with advanced technology. The author visited Showa Engineering in

Tokyo in November 1992 and received their assistance with two plant evaluation exercises.

Sumitomo Seika Chemical Company Limited (a division of the Sumitomo group and apart from Mitsubishi, Sumitomo is the second largest business organisation in Japan) have their own in-house oxygen PSA technology (Sumitomo Seika, 1990). They have built and commissioned the largest oxygen PSA plant in the world to date, and have exported several plants to Europe. Sumitomo Seika is a major PSA plant supplier and has recently been involved with Air Products and Chemicals, an American company represented in South Africa.

2.3.4 BOC Group Technology

Access to the British Oxygen Company group technology was obtained through Afrox Limited. This technology is currently being developed by the PSA Oxygen Centre of Excellence, based at Guildford in the United Kingdom. This PSA Centre of Excellence has been established to monitor global oxygen PSA developments, to coordinate any PSA project undertaken by a BOC group operating company and to distribute the plant results to the rest of the BOC group. This technology is developing rapidly and major technological advances have been achieved in the last eighteen months, during the period of this investigation. The BOC group does not have a large number of reference plants, however BOC technology appears to be very comparable to that of the major world leaders.

2.3.4.1 Mainline Equipment

BOC will issue any of its group operating companies requiring a PSA plant with an provisional outline process design package. This package will be a design proposal only, with vessel sizes, air flow requirements, valve sequencing programmes and control philosophy. It is up to this BOC group operating company to assess this package and produce a detailed plant design. This design will then be added to the standard range of plants throughout the BOC framework. The pilot plant mentioned in this text is built to verify a provisional BOC 10 TPD process design package.

BOC have provisional plant process design packages available for both pressure vacuum and pressure atmospheric plant cycles. The feed air pressure and flow would be supplied by a locally sourced low pressure blower, either Roots or centrifugal type, or a high pressure supply would be met with an oil free screw compressor. Vacuum equipment, if required is to be sourced in a similar manner. This eliminates excessive amounts of imported plant spares to be held in stock for any imported equipment. BOC has a patent on an air drying system contained within the adsorber vessels on a two bed PSA plant that involves an activated alumina layer to dry the feed air before it contacts the zeolite molecular sieve. This eliminates the need for a refrigerant air dryer and leads to an overall plant capital cost reduction.

Pressure vessels and pipework are all designed to the relevant prevailing engineering codes. In South Africa, ASME VIII or BS codes would be preferred as they are commonly used and accepted by the Department of Manpower (MOSAct, 1992). These codes incorporate strength calculations for cyclical loading and monitored stress relieving.

Process actuated valves are sourced from local agents representing overseas companies. Several South African valve manufacturing companies including IPV and Bestobell were contacted regarding high performance butterfly valves, but few manufactures were prepared to guarantee these valves to perform one million cycles. For this reason it is preferable to use tried, tested and guaranteed imported valves types.

The plants will be built on site by a local project engineering team. This ensures that strict group standards relating to Total Quality Management and oxygen cleanliness are met. To eliminate any potential plant operating risks, every new plant that is to be installed within the BOC group must have a complete Hazard Operating (HAZOP) study performed, which will identify any potential problems before they present themselves in an operating environment.

Product compression is usually supplied by using an oil free reciprocating oxygen compressor. A patented liquid ring oxygen compressor for use on all types of oxygen PSA systems is currently being jointly developed in South Africa in conjunction with Siemens. The liquid ring compressor is a low cost unit capable of being suitably modified with stainless steel impellers and bronze crash plates to prevent sparks being formed during a possible machine failure situation in an oxygen enriched environment.

Throughout the entire project period, the BOC oxygen PSA Centre of Excellence will provide technical support and recommendations on the use of mainline equipment. All recommendations made would be incorporated in the BOC design packages used in future plant designs. The BOC oxygen PSA Centre of Excellence would also be prepared to assist during the plant commissioning if their assistance is required.

2.3.4.2 Cycle Efficiency

The BOC group have produced design packages with a calculated theoretical power demand. These figures have not yet been proved, as the BOC group has only built a few reference plants to date. Computer models of expected BOC plant performance indicate that these plants are expected to compare very favourably with other leading PSA manufacturers. The BOC three bed VSA process cycle uses a power demand slightly higher than most Japanese plants and the capital cost of the BOC installation is more favourable, due to smaller vessels being used with a molecular sieve that incorporates a high specific product and therefore a higher product recovery.

The BOC two bed PSA process cycle was developed in May 1992 and the theoretical power figures calculated are similar to most other manufacturers, but these performance figures have not yet been proved. These figures will be verified on the completion of two plants that are scheduled to be commissioned in Nigeria and India during the course of 1993.

2.3.4.3 Plant Outputs

All of the BOC plants, both PSA and VSA, conform to the plant outputs for the Pressure Atmospheric and Pressure Vacuum cycles given in sections 2.3.1.3 and 2.3.2.3 respectively.

The BOC group companies intend to design and build plants to accommodate the balance between capital costs and plant operating efficiencies, thus keeping abreast with leading technological developments for oxygen PSA plants.

2.3.4.4 Manufacturers

Numerous companies could be contracted by BOC to build PSA plants on their behalf. Most of these companies are subsidiaries of the BOC group operating companies. Any local project management company could manufacture a PSA plant under suitable guidance from a specialist PSA Engineer. A pressure vessel manufacturing company called Industrial Research and Development has been contracted to build the vessels and interconnecting process pipework during the pilot plant construction as discussed in section 4.3.1. Any such organisation could complete the construction of a PSA plant under suitable guidance. A PSA Engineering specialist would usually be required to assist with the plant start up, commissioning and performance optimisation.

3 THEORETICAL CONSIDERATIONS

3.1 GENERAL

3.1.1 Principals of Adsorption Separation

Separation may be defined as a process that changes a mixture of substances into its constituent parts that differ with each other in composition. Separation is a process that is the opposite to that of mixing.

The surface of a solid body represents the boundary of its structure. Unsaturated forces acting on this surface will cause a gas flowing over the surface to attach and form bonds. This action is called adsorption. The development of modern adsorbents and process cycles has led to major advances in separating atmospheric gases.

The development of synthetic zeolite, (or molecular sieves), led to an adsorbent that would preferentially adsorb nitrogen over oxygen by a factor of approximately three. (Nitrogen is four times more abundant than oxygen in the atmosphere). This adsorbent characteristic is used in a process that will separate oxygen from the atmosphere. This process has to be regenerative to permit the re-use of the molecular sieve in further cycles as well as allowing recovery of the adsorbed gas. Efficient pressure swing cycles were invented concurrently by Skarstrom and by Geurin de Montgareuil and Domine (Ruthven, 1984: 361). Since then, numerous refined and sophisticated pressure swing cycles were developed, making significant improvements on the separation efficiencies and process energy requirements.

While commercial developments were taking place, fundamental theories are being written about separation processes. Since 1970 many significant theories have been developed on the equilibrium adsorption of mixtures on pure gas isotherms. Recently, from 1985 onwards, publications such as Gas Separation and Purification have appeared describing theories for the adsorption separation of mixtures, typically based on Langmuir linear isotherms. This has led to computer modelling and simulation of cyclical pressure swing processes.

3.1.2 Adsorption Process

Adsorption is the fixation of gas molecules on the surface of a solid body without any reaction between the molecules of the solid body and the adsorbed gas. When an equilibrium occurs between a gas phase and solid surface (adsorbent and adsorbate equilibrium), the gas molecule concentration is greater near the solid surface than in the rest of the gaseous mass. The phenomenon leading to this distribution of concentration is called Van der Waals adsorption or physical adsorption. It is therefore useful to develop in a molecular sieve the greatest possible internal area using capillaries and pores in the zeolite crystal structure.

Adsorption increases with the relative pressure of the adsorbate. This variation is represented at a constant temperature by the isotherm of adsorption. Zeolite molecular sieves, used for the separation of air, have a high adsorption capacity at low relative pressures. Adsorption decreases when operating temperatures increase. It is therefore important to maintain the feed air temperature well within the operating temperature range of the specific molecular sieve.

Quite simply, when a flow of air is passed over a molecular sieve bed, only the molecules with a diameter smaller than the opening of the molecular sieve pores will enter and be adsorbed. The diameter of oxygen and nitrogen molecules are approximately 2.5 and 3 Angstroms respectively. A molecular sieve used to adsorb nitrogen will also adsorb a small amount of oxygen, however, the preferential adsorption of nitrogen will be about three times to that of oxygen.

The ease of air separation is determined by the relative volatility of its components, which is simply the ratio between the vapour pressures of the oxygen and nitrogen.

The parameter used to define adsorption between any two gases, i and j, is the separation factor, (α), defined by :

$$\alpha_{ij}=\frac{X_i/Y_i}{X_j/Y_j}$$

where X_i and Y_i are the equilibrium mole fractions of a component i in the adsorbed and the gas phases respectively and X_j and Y_j are the equilibrium mole fractions of component j in the adsorbed and the gas phases respectively. (Ruthven, 1984: 3).

To decide when to use adsorption separation or cryogenic distillation separation, the following factors should be considered :

- The relative volatility (ratio between the vapour pressure of both components of the air, being oxygen and nitrogen) between the components should be between 1,2 and 1,5.
- The majority of the feed gas is of a relatively low value, more volatile constituent (eg. nitrogen) and the required constituent (eg. oxygen) is in a relatively low concentration. This will cause a large reflux requirement on a distillation column and would therefore favour adsorption.
- The constituents to be separated should contain geometrically or chemically dissimilar molecules.
- A low cost, low purity product is required.

A high separation factor favours adsorption. For oxygen and nitrogen the relative volatility is fixed and the separation factor will vary widely, depending on the choice of adsorbent. The first requirement for the efficient design of an oxygen PSA system lies with the choice of adsorbent. This outlines the difference in the process design parameters of the two PSA plant types, being the two bed PSA cycle and the three bed VSA cycle.

3.1.3 Adsorbent Regeneration

In order to ensure good performance and maximum possible plant life, molecular sieves must be regenerated, i.e. brought back to their previous state. Experience shows that there is no desorption hysteresis (changes caused by magnetic and electrolytic influences) and that the regenerated molecular sieves are identical to new ones. Aging or decay of the molecular sieve adsorption and mechanical properties is very slow. There are two fundamental methods of regenerating the molecular sieves. The first method is by changing the isotherm and is known as thermal swing adsorption, or TSA and it is not normally used for separation of oxygen from air. The second method is by changing the relative pressure and is known as pressure swing adsorption, or PSA. This is the process that has been developed for the separation of atmospheric gases.

The main principal of pressure swing adsorption is to retain the heat of adsorption inside the bed and to keep it available as desorption heat. A small amount of product oxygen from an on stream adsorber vessel is expanded into the desorbing bed. This gas divides its relative pressure by the cycle operating pressure ratio. The resulting purer gas is vented counter currently through the regenerating vessel and thus removes the adsorbed gas, nitrogen. This process is known as a purge step.

3.1.4 Adsorbent Characteristics

The most important adsorbent characteristic is its high porosity. Due to this, the physical characteristics are generally more important than the chemical structure.

PROPERTY	TEST	RESULT
Bulk Density		640 kg/m^3 minimum
Residual Water	Karl Fischer	1,1 wt% maximum
Crush Strength		1,8 kg minimum
Bead Size	<3,35 mm	100% minimum
	<2,36 mm	95% minimum
	<1,70 mm	10% maximum
	<1,40 mm	5% maximum
	<0,85 mm	0,5% maximum
Pore Volume at 77K, 75 mm Hg	O_2 Capacity	32,5 wt% minimum
Nitrogen uptake	297K, 700 mm Hg	2,63 wt% minimum

TABLE 3.1

TYPICAL SORBENT CHARACTERISTICS

(UOP type Cax VSA II Molecular sieve)

The important physical characteristics are pore volume, pore size distribution, surface area, crush strength, bulk density and erosion resistance. Table 3.1 outlines typical sorbent characteristics for a type 5A molecular sieve (UOP Molsieve, 1992), as used to separate oxygen from air. This sieve would thus adsorb the nitrogen molecules and allow the oxygen molecules to pass through as product gas.

The surface area of a sorbent can be determined by the Brunauer Emmett-Teller (BET) equation. This is used to calculate the amount of nitrogen for monolayer coverage. The surface area is taken for the monolayer coverage based on the nitrogen molecular area taken as 16,2 Å^2 (Brunauer, 1943) by assuming that the nitrogen is in the liquid state the molecular sieve structure is that of hexagonal close packing.

Total specific pore volume and its distribution over the pore diameter are needed to assess the average pore volume. Helium, because of its small atomic size and negligible adsorption, fills the total voids and gives the accurate pore volume. The size distribution is measured by nitrogen adsorption for larger molecular sieve pores in the range from 10Å to 250Å.

The pore radius, (r), at a given pressure (P) is calculated by a balance between the contact angle (Θ) and the interfacial tension (σ) :

$$r=-\frac{2\sigma Cos\theta}{P}$$

Generally σ is taken as 0,48 N/m^2 and Θ has an average value of 2,44 radian (140°) resulting in r = 7500/P (Yang, 1987: 11). The nitrogen adsorption technique takes advantage of this phenomenon where capillary condensation occurs at a relative pressure below unity because the equilibrium vapour over a concave meniscus is lower than that over a plane surface (P_o), as stated in the Kelvin equation (Yang, 1987: 11)

$$\ln\frac{P}{P_o}=\frac{-2\sigma V_m Cos\theta}{r_k RT}$$

From an adsorption uptake curve of nitrogen at 77 K, the pore size distribution can be calculated on the basis of the Kelvin Equation.

3.1.5 Zeolite

During the eighteenth century, a Swedish chemist observed that various minerals appeared to melt and boil when heated, with a considerable release of water vapour. He called these minerals Zeolites, a word derived from 'zein', (to boil) and 'lithos', (stone). Experimental work performed on these minerals in the 1950's led to the development of synthetic zeolites and molecular sieves (Rhone Poulenc, 1989). Further development in the 1970's led to the manufacture of molecular sieves that would be suitable for the separation of oxygen from atmospheric air.

Zeolites, also known as sorbent, adsorbate or molecular sieves are artificial sorbent materials manufactured from crystalline alumina silicates of alkali or alkali earth metals such as sodium, potassium and calcium. Stochiometrically, zeolites are represented as :

$$M_{x/n}[(AlO_2)_x(SiO_2)_y]zH_2O$$

where x and y are integers with $x/y \geq 1$, n is the valence (energy level) of cation M (added to change pore size) and z is the number of water molecules in each unit cell (Union Carbide, 1987). The cations balance the electrical charge of aluminium atoms, each having a charge of -1. The water molecules can be removed by heating or evacuation to leave an unaltered aluminosilicate skeleton with a void fraction between 0,2 and 0,5 (Yang, 1987: 20). The size of the window apertures (the open spaces of the crystal structure) can be controlled by fixing the type and number of cations, ranging from 3Å to 10Å. This gives sorption selectivity and defines the pore size which will allow the preferential adsorption of certain molecules.

There have been many types of synthesized zeolites developed. They are designated by letters, for example; type X, type Y, type ZSM and type MSC. Type 5A is used for nitrogen adsorption when oxygen is the required product gas. The difference between type X and type Y sieve lies in the Si/Al ratio, which is typically within the range of 1,0 - 1,5 for X and 1,5 - 3,0 for Y (Ruthven, 1984: 10). The difference between the type A and the type X molecular structure lies in the manner in which the truncated octahedrons are joined. The truncated octahedrons shown on Figure 3.1 is a cube with square and hexagonal sides. To form type A zeolite, the truncated octahedra are joined by their square sides, as shown in this figure. To form a type X molecular sieve the truncated octahedra are joined by their hexagonal sides.

There is a corresponding difference on the number of exchangeable univalent cations for the various types of molecular sieve. This varies from about 10 to 12 cations per cage for type X and down to as low as 6 cations for high silicon type Y molecular sieves (Ruthven, 1984: 13).

The molecular structure of type A zeolite (used for nitrogen adsorption) is based on truncated octahedra as a base unit. The main cell is based on 8 sodalite cages, located at the corners of a cube and joined through four membered oxygen rings, forming a molecular structure known as 4SR. This arrangement forms a large polyhedral cage with a free diameter of about 11,4Å (Yang, 1987: 21), accessible through eight membered oxygen windows. Stacking these units in a cubic lattice gives a three dimensional isotropic channel structure, constricted by the eight membered oxygen rings.

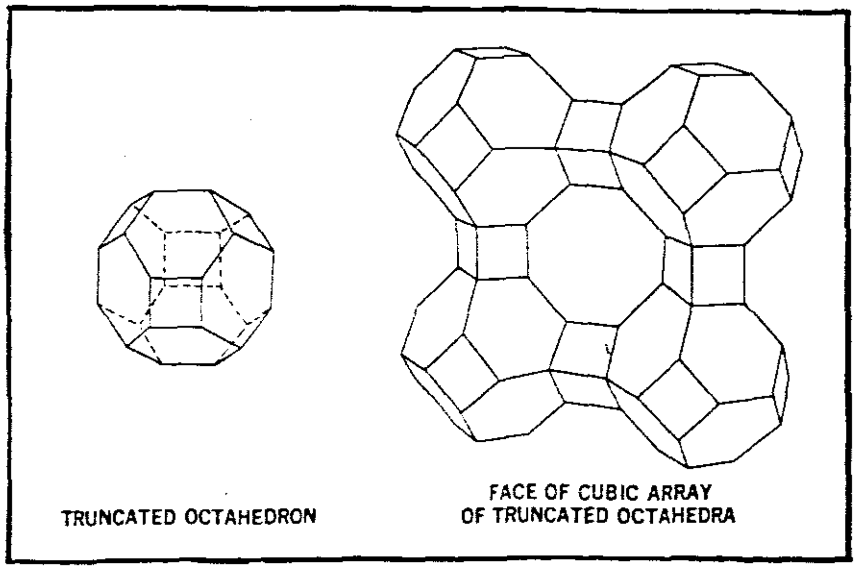


FIGURE 3.1

TYPE 5A TRUNCATED OCTAHEDRONS (UNION CARBIDE,1987)

Each base cell contains 24 tetrahedral (AlO_2 or SiO_2) units and as the Si/O_2 ratio in zeolite A is always close to 1.0 there are 12 univalent exchangeable cations per cell. Three distinct cation sites have been identified. These cation sites are the places on the cell where the cations are most likely to be found. These are near the centres of the six rings in the eight corners of the central cavity (type I), in the centres of the eight rings (type II) and on the cage wall in close proximity to a four-ring (type III). With most cations, the type I sites are preferentially occupied, followed by type II sites and the type III sites are filled only after type I and II have been occupied. In the sodium (type 4A) form, there are 12 cations per cage. These are found in the 8 type I sites and 3 type II sites (the 6 eight-rings are each shared by two cages) with one cation in a type III site. All windows are partially obstructed by a sodium cation, reducing the sieve aperture from about 4,4Å to 3,8Å (Yang, 1987:21). If Na^+ cations are exchanged for Ca^{2+} or Mg^{2+} the number of cations per cell decreases. At 67% exchange there are only 8 cations per cell and these can be accommodated in type I sites. Thus in Ca^{2+} or Mg^{2+} (type 5A) the effective aperture is increased and larger molecules can penetrate. A type 3A sieve is obtained with potassium exchange. The diameter of the potassium ion is greater than that of sodium and therefore a sieve with a smaller aperture is produced, as the potassium ion occupies more space than the sodium ion.

3.1.6 Adsorbent Selection

The selection of a sorbent for any application is often a complex problem and this is usually based on the equilibrium isotherm. The equilibrium isotherms of all constituents in a mixture must be considered at the operating pressures and temperatures of the cycle. Based on these isotherms the following factors need to be considered :

- The adsorbent capacity, at the operating temperature and pressure;
- The method of sorbent regeneration ie, Pressure or Temperature Swing;
- The length of unusable bed (LUB);
- The required product purities.

The LUB is approximately 0,5 times the span of the concentration wave front (Yang, 1987), or the mass transfer zone. A short LUB is desired because it results in a high sorbent productivity and high product purity. Pore size distribution and the equilibrium isotherm both contribute to the length of unused bed.

The separation of oxygen and nitrogen from air is based on kinetic separation, being the difference between pore diffusivities of two gases and selective molecular exclusion.

A low total void space is required for high product recoveries, since the gas mixture remaining in the void space in the saturated bed is usually not recovered as useful product.

The selection of a molecular sieve is usually undertaken by the process design authority and the molecular sieve manufacturer. In the cases of the pilot plant discussed in this text, BOC is the process design authority and UOP is the sieve manufacturer. Various other molecular sieves are suitable for this type of process and these include Bayer Baylith sieve (Bayer, 1991), Union Carbide sieve (Union Carbide, 1987) and Rhone Poulenc Procatalyse sieve (Rhone Poulenc, 1989).

3.1.7 Equilibrium Adsorption

For a given solid gas pair, the amount adsorbed at equilibrium is given by :

$$v = f(P,T)$$

At a fixed temperature, T, the volumetric amount adsorbed, v, is only a function of total pressure, P. This is defined as an adsorption isotherm (Yang, 1987: 26).

There are two types of bonding between the adsorbate molecule and the solid surface, namely, physical (or kinetic) adsorption and chemical adsorption.

Chemical adsorption involves electron transfer forming a weak bond and is an easily reversible process. This process is not needed for oxygen production from atmospheric gas separation but it can be utilised to adsorb oxygen in a nitrogen PSA system.

Bonds formed by physical adsorption are held by dispersion and repulsion forces, commonly known as Van der Waals or electrostatic forces, originating from ionic atoms and polar groups on the molecular sieve surface. The surface area of the adsorbent possesses a degree of polarity. The capacity of the sorbent depends on the surface area and the sieve porosity. The driving force for all gas separation processes lies in the departure from the equilibrium isotherm. Information must be known on the adsorption isotherm for the design of the process cycle.

3.1.7.1 Isotherm Models

Many theories and models have been developed to predict adsorption on the limited number of experiments undertaken. The resulting isotherm equations are based on three different approaches (Yang, 1987: 27) :

- The Langmuir approach : Langmuir in 1918 originated the Kinetic approach, assuming the system adsorption to be equal to that of condensation. The Langmuir isotherm is still the most useful isotherm for adsorption separation that is currently being used, especially for atmospheric gas separation.

- The Gibbs approach : The Gibbs adsorption isotherm is defined as :

$$-Ad\pi + nd\mu = 0$$

  This assumes a two dimensional equation of state of the adsorbed film, relating π-A-T. An integration of the Gibbs equation will give the required isotherm. There are always the same number of isotherms as equations of state.

- The Potential Theory: This assumes a gradual concentration of gas molecules near the solid surface due to a potential field. This relationship, called a characteristic curve, is based on the potential field and the volume above the surface and is independent of temperature. This is mainly used with Molecular Sieve Carbon (MSC) type sorbents and is applied to the adsorption of oxygen in a nitrogen PSA production process.

3.1.7.2 Adsorption Forces

The forces involved in physical adsorption include both Van der Waals forces (dispersion and repulsion) and electrostatic interactions comprising polarisation, dipole and quadrapole interactions. The dispersive forces exist between any two atoms or molecules, one being in the adsorbate and the other being on the surface of the adsorbent. These forces arise from the rapid fluctuation of the electron density of each atom, which induces an electrical moment in its neighbour and thus creates an attraction between them.

The potential energy is approximated by (Yang, 1987: 28) :

$$E(r) = -Cr^{-6}$$

The negative sign denotes attraction. The short range repulsion potential is approximated by (Yang, 1987: 28) :

$$E(r) = Br^{-12}$$

Therefore the total potential energy is :

$$E(r) = -Cr^{-6} + Br^{-12}$$

This is shown in Figure 3.2

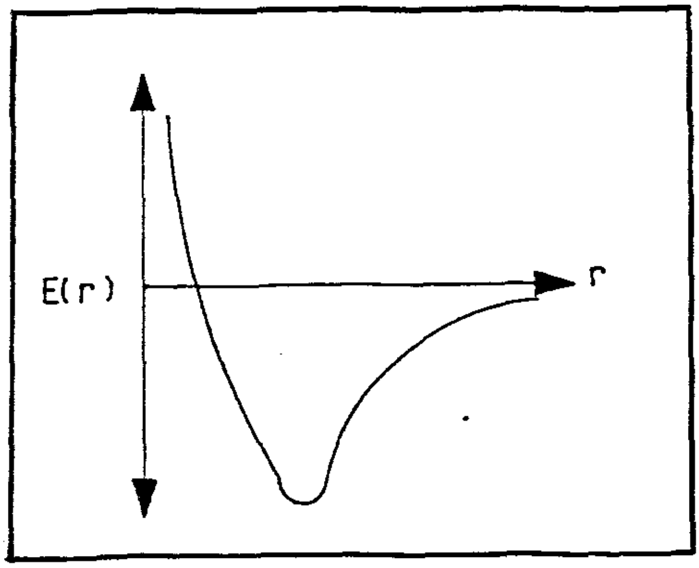


FIGURE 3.2

THE POTENTIAL ENERGY BETWEEN ATOMS SEPARATED BY DISTANCE (r).

The dispersion constant, C can be calculated theoretically by a complex relationship between the speed of light, the molecular polarizabilities and the magnetic susceptibilities of the two approaching atoms (Yang, 1987:29).

The potential energy function, (ϕ) between the centres of a gas molecule and the plane going through the surface atoms, can be found by summing all interconnecting pairs of atoms.

$$\phi(z)=-\sum_i(C_{ij}\sum_j r_{ij}^{-6})+\sum_i(B_{ij}\sum_j r_{ij}^{-12})$$

For a polar surface, including a dipole in the adsorbate molecule, the interaction energy is (Yang, 1987: 29) :

$$\phi_p=-\tfrac{1}{2}\alpha^2F$$

If the adsorbate molecule has a permanent dipole, the additional energy is (Yang, 1987: 30) :

$$F_\mu = -F\mu Cos\theta$$

When considering the adsorption forces, the bond strength should be higher on a more polar surface for a given adsorbate molecule. For the same surface the bond strength is higher for gas molecules with permanent dipoles and quadropoles.

3.1.7.3 The Langmuir isotherm

The Langmuir isotherm is the most useful isotherm for kinetic adsorption and is based on the following :

- Molecules are adsorbed at a fixed number of well defined local sites;
- Each site can hold only one adsorbate molecule;
- All sites are equally energetic;
- There is no interaction between molecules adsorbed on neighbouring sites.

The Langmuir isotherm is based on the concept of dynamic equilibrium between the rate of adsorption and desorption.

Occupied sites are no longer available for adsorption, therefore the rate of adsorption per unit surface area is (Yang, 1987: 31) :

$$v=\frac{P}{(2\pi mbT)^{1/2}}$$

The rate of desorption per unit surface area, at dynamic equilibrium (Yang, 1987: 31):

$$B=\frac{\alpha}{\beta(2\pi mbT)^{1/2}}-e^{Q/RT}$$

B is known as the Langmuir constant. This fits the type of isotherm necessary for nitrogen adsorption and henceforth oxygen production. The temperature dependence of B is:

$$B=e^{Q/RT}T^{-1/2}$$

As Q is always positive its value decreases with increasing temperature, because physical adsorption is always exothermic. The free energy must decrease in an adsorption process. Entropy decreases due to the decrease in the number of degrees of freedom of the gas molecule in the occupied site and the enthalpy change must therefore be negative. This is defined as (Yang, 1987: 31) :

$$\Delta G=\Delta H-T\Delta S$$

The resulting effect of the free energy change implies that there should be a cooling effect created in the desorbing gas stream. In this case of oxygen production, a small decrease in temperature is expected in the waste nitrogen stream that is being desorbed during the molecular sieve regeneration steps. This cooling effect could also present itself in the form of a cold spot contained within the molecular sieve bed. These cold spots could reach temperatures well below freezing point. Cold spots should not be detrimental to the gas separation process in any way.

3.2 PSA TECHNOLOGY

3.2.1 Theoretical process cycles

The goal of research in the 1950's and 1960's as well as the original invention of the PSA process was oxygen separation from atmospheric air. The original ideas of separating air by pressure swing adsorption were patented individually in 1958 by two parties. Skarstrom filed a patent in which the Skarstrom cycle purges the saturated sorbent bed at a low pressure by using a fraction of the light product. Guerin-Domine filed a similar patent, in which the Guerin-Domine cycle used a method of vacuum desorption.' Both of these pressure swing cycles yielded modest separations of oxygen due to the low separation factors between atmospheric gases, usually between two and three on zeolite. Since their initial conception in 1958, numerous modifications to these cycles have been made. Today they are now commonly known as the Pressure Swing Adsorption cycle (PSA) and Vacuum Swing Adsorption cycle (VSA).

The actual commercialization of the PSA oxygen process was only realised in 1970. Because of the low adsorption selectivity of nitrogen and argon, air separation is considerably more difficult than air drying and hydrogen purification in the Petro-chemical industry. As shown in Figure 3.3 the selectivity ratio of oxygen to nitrogen (the ratio at which one component is preferentially absorbed) on 5A Zeolite is between two and three, which is in the typical pressure range (shown in atmospheres) of practical operation. The loading weight percentage, (wt %) is the unit mass of nitrogen that can be absorbed per unit mass of sieve. As seen in Figure 3.3 argon is also obtained as an extra product along with oxygen, thus limiting the purity for the oxygen product to approximately 95% since air contains 0.934% argon by volume.

3.2.2 Pressure swing adsorption

The Skarstrom Cycle shown in Figure 3.4 uses a two bed apparatus. The original patent was assigned to the Esso Research Company. This was used for hydrogen purification and feed stream drying in the Petro-chemical industry (Yang, 1987: 238).

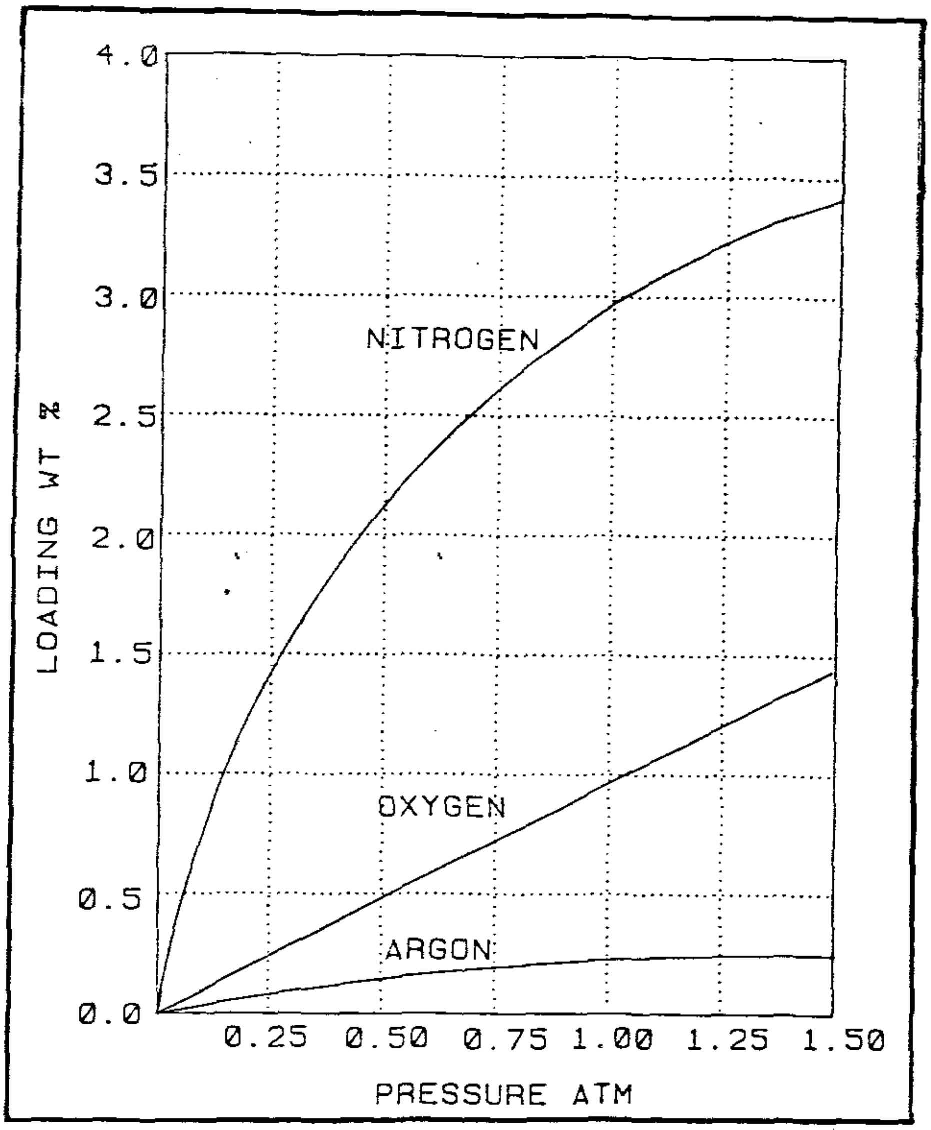


FIGURE 3.3

TYPE 5A MOLECULAR SIEVE ISOTHERMS SHOWN AT 25°C

Two steps are involved in the basic Skarstrom cycle for each bed. The first being the re-pressurization followed by feed in bed A, while oxygen is withdrawn. The second is blowdown and purge with oxygen of B, whilst nitrogen and waste gases are withdrawn. The functions of beds A and B are reversed in the next cycle.

The first major process improvement was the introduction of the co-current depressurization step. To incorporate this step, the adsorption period is stopped well before the break point, which is when the mass transfer zone (or concentration wave front) is far from the outlet of the bed. The adsorptive step is immediately followed by a co-current depressurization before the bed is desorbed by further blow down and purge. The major function of this co-current depressurization is to increase the concentration of the oxygen in the bed by increasing the relative pressures of the oxygen in relation to the nitrogen. The nett result of co-current depressurization is oxygen product purity enhancement. This co-current depressurisation step is more commonly known as purge.

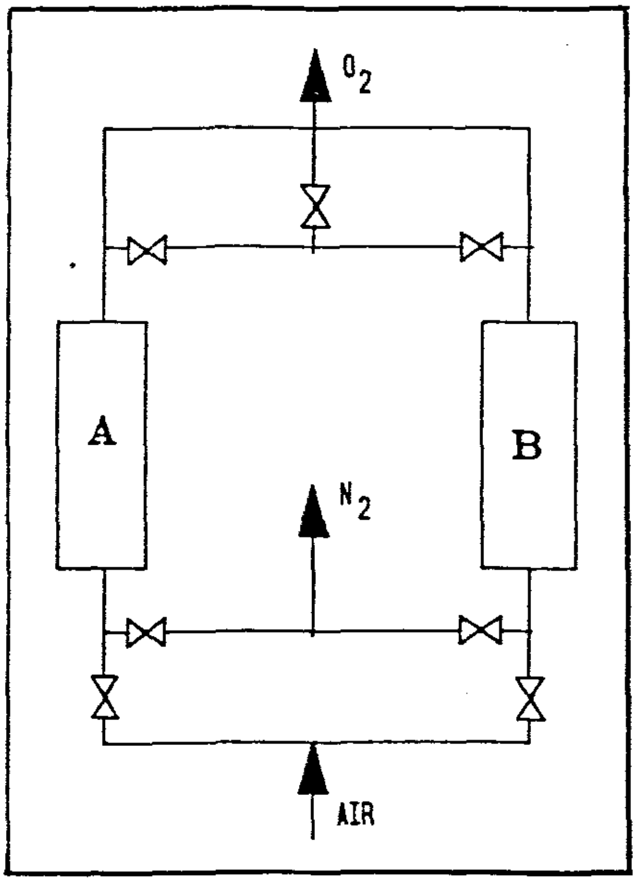


FIGURE 3.4
THE SKARSTROM CYCLE

3.2.3 Vacuum Swing Adsorption

The Guerin-Domine Cycle shown in Figure 3.5 usually uses a two bed apparatus. The original patent was assigned to L' Air Liquide in France (Yang, 1987: 239).

This cycle is a versatile one. Depending on the nature of the mixture to be separated, it can vary in the number of beds, the bed interconnections and the scheme of operation. Designs from one to six beds have been proposed. For a two bed system as shown in Figure 3.5, each bed goes through three steps:

- pressurization (with only the feed end open);
- depressurization through the other beds, both in a co-current direction to the feed when oxygen is formed;
- and evacuation from the midpoint of the bed when nitrogen is formed.

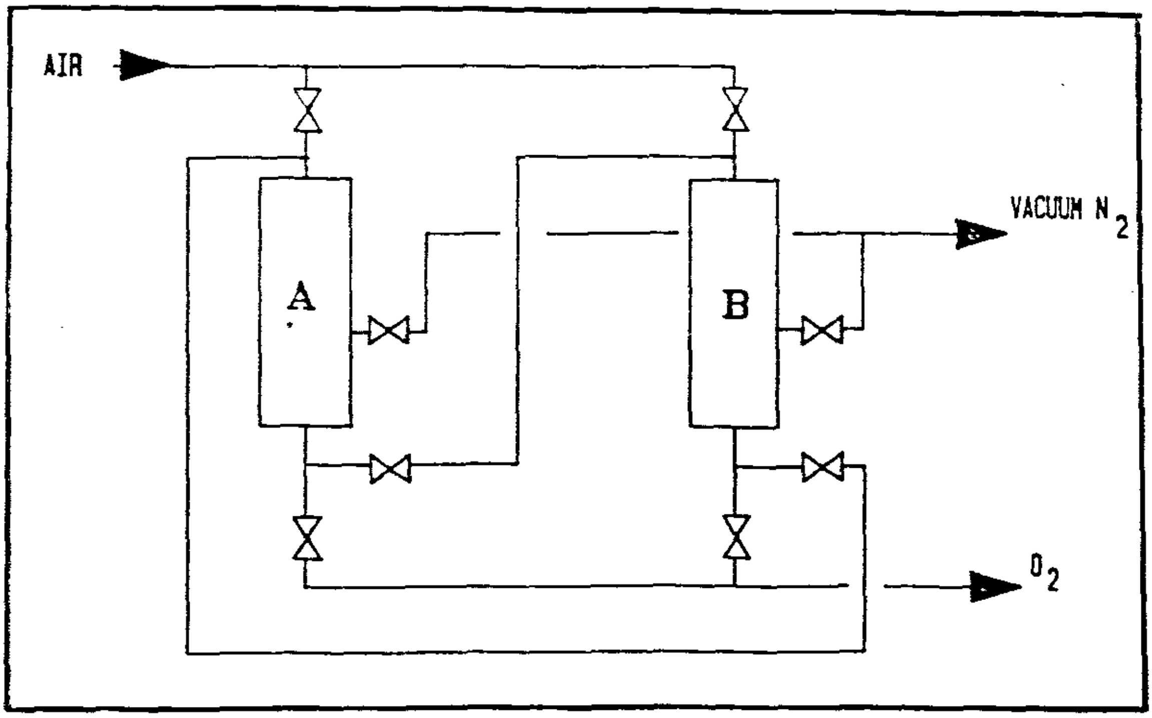


FIGURE 3.5
THE GUERIN DOMINE CYCLE

Using 5A Zeolite in the Guerin-Domine Cycle, excellent results were obtained on air separation. The separation purity results were 98% oxygen (argon free) and 96,5% nitrogen, at 51% and 58% product recovery respectively. These results were substantially better than Skarstrom's in both product purities and recovery. Because of the low selectivity between oxygen and nitrogen, a good separation was obtained with the use of the evacuation step to desorb the bed. The short cycle time kept bed temperature fluctuations to a minimum.

With improvement to this cycle, such as co-current depressurization, pressure equalization and backfill, various manufacturers have managed to successfully commercialise the vacuum swing adsorption process since 1970 for simultaneous oxygen and nitrogen generation. The pilot plant discussed in section 3.3 is based on a modification of the Guerin-Domine cycle.

3.2.4 Commercial Processes

In 1983 there were 200 PSA units installed worldwide between 1 and 36 TPD. These systems are essentially a modification of the Skarstrom cycle. Vacuum desorption is used in some applications. Three to four beds are also generally used. The four bed system is identical to that used in hydrogen purification and an outline of the process is shown in Figure 3.6. These systems generate 90% to 95% oxygen with between a 30% to 60% product recovery. The compressed air is pre-cooled to 30°C and fed to the plant at approximately 1250 mbar (abs.). Liquid moisture condensate is removed in coalescing filters and pre-treatment beds of activated alumina.

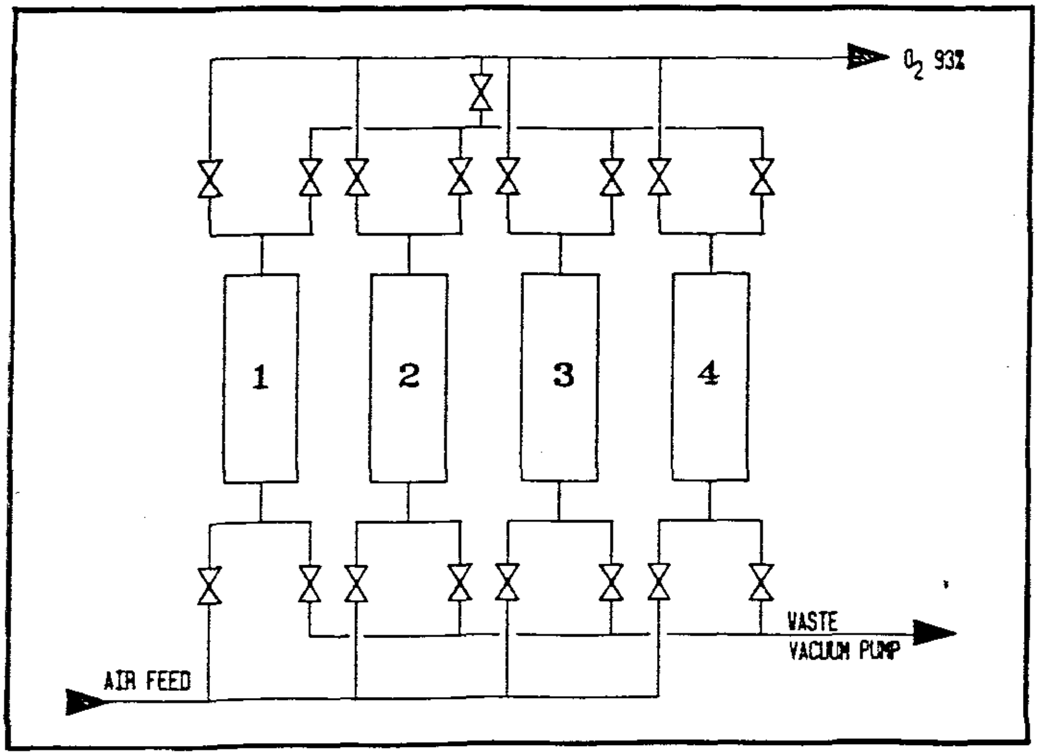


FIGURE 3.6
FOUR BED PSA SYSTEM (SHOWA, 1991)

Small units for medical oxygen were commercialized in the mid 1970's. They are two bed PSA systems, each containing 1 to 2 kilograms of zeolite, which operate on the original Skarstrom Cycle. The product purity varies between 85% and 95% at only a 10% to 30% oxygen product recovery. These systems have a relatively low energy requirement. The process is similar to that outlined in Figure 3.4, as they tend to follow closely to that of the Skarstrom cycle.

3.2.5 Model for Skarstrom Cycle

The production of oxygen from air on a two bed PSA system makes use of the Skarstrom cycle. The simple Skarstrom cycle is shown in Figure 3.7, as a two bed PSA Cycle.

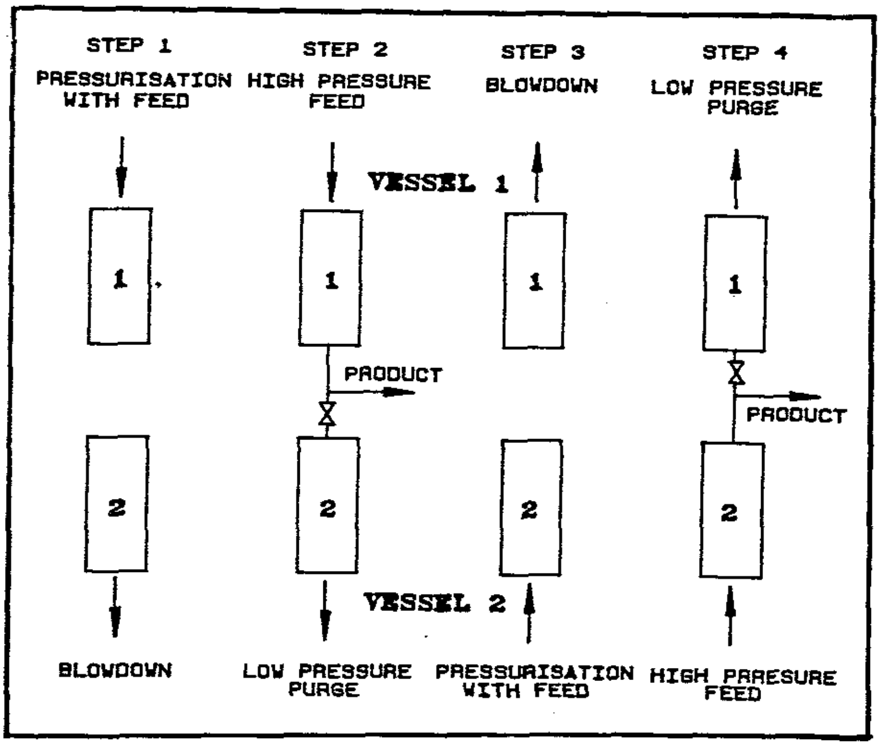


FIGURE 3.7
SKARSTROM CYCLE ANALYSIS

This cycle combines the two steps of bed pressurization with adsorption, feed introduction and counter-current blow down and purge. In actual production, four steps are used and each step introduces a concentration wavefront into the bed. Assuming an ideal linear isotherm, all wave fronts will retain their shape, while passing through the adsorption column for both adsorption and desorption. The wavefront velocities can be calculated from sieve characteristics and assumptions of ideal gas behaviour. The average compositions of the gas mixtures behind the wave front can also be calculated. The waste gas concentration can be calculated as a weighted sum of the concentrations behind the wavefront. To solve the model of the Skarstrom cycle, it is necessary to locate the gas characteristics in a time-distance plane and to count the number of changes in slope that each characteristic experiences. To solve the model, the following assumptions are also made:

- Linear isotherms are followed by both components (named A and B). The strong adsorptive, A is at trace level. The two isotherms do not interfere with each other;
- The cycle is isothermal, i.e. constant temperature;
- The bed flow velocity is constant during the adsorption and purge steps;
- The heat and mass transfer are instantaneous;
- Plug flow (laminar) is assumed, with no axial or radial dispersion;
- Ideal gas laws are obeyed;
- The pressure drop across the bed is negligible.

Both Yang (1987) and Ruthven (1984) offer complicated solutions to the Skarstrom cycle. This cycle analysis follows the point of a mass balance, whereby the independent adsorption of each component, A and B, and their desorption later must be equal. From the adsorption rates, the velocity of the concentration wavefront and the penetration depth can be determined. Furthermore, the critical purge to feed ratio can be determined to optimise the molecular sieve regeneration process. For complete purification, a breakthrough of feed gas into the product does not occur and the purge to feed ratio must be greater than a defined critical value to maintain a positive penetration depth. The concentrations and flow rates of the wastes from all the process steps can be calculated by determining the gas characteristics with respect to time and summing the waste functions. The critical purge to feed ratio varies with respect to the pressure ratio and is inversely proportional to the separation factor. The Skarstrom cycle also shows a maximum product recovery with a pressure ratio below four.

3.3 PILOT PLANT DESIGN

Various factors influence the design of PSA plants. The process design is a chemical engineering function, briefly explained earlier in this section. Of interest to this thesis, are the mechanical engineering aspects required to provide the operating parameters to which this process is designed. The plant design, as detailed in this section, is for that of a BOC designed plant, built as a pilot plant and is the first operational plant in South Africa that produces oxygen from the atmosphere by utilising a pressure swing adsorption technique that involves a vacuum pump to regenerate the molecular sieve beds.

During November 1991, as the Author was employed to study all aspects of PSA, it was decided that it would be advantageous to build a pilot 3 bed oxygen VSA plant. This was to be used as a pilot plant, to enhance the local understanding of PSA processes and to serve as a technology demonstrator to South African Industry. It was also expected that useful performance and operational data could be obtained from this plant.

In determining the size of this plant, factors such as transport, lead time of major plant equipment, marketing requirements and design criteria were considered. The largest transportable plant was required, to easily transport the plant from a test site to an operational site and the plant should easily meet the needs of a specific application. Following this, a pilot plant of 10 TPD oxygen was to be built. The estimated cost structure for this plant is tabled in Appendix 1.

3.3.1 Process Design

The choice of a theoretical process cycle will depend upon the size of plant to be built. The 2 bed pressure atmospheric cycle can be used in a plant from a few kilograms per hour to approximately 30 TPD, whereas the 3 bed vacuum cycle can be used from about 5 TPD to 130 TPD. Once the size of the plant has been determined and the theoretical process has been chosen can the chemical design be completed.

The chemical process design is always based on the choice of sorbent. This has been briefly discussed in section 3.1.6. Various types of molecular sieves are commercially available. All sieve manufacturers treat information on molecular sieve characteristics as strictly confidential. The choice of sorbent will determine the plant pressure and pressure ratios to achieve effective adsorption and regeneration of the sorbent. This will usually follow a particular adsorption isotherm at a fixed temperature, which must correspond with the air feed temperature. The selected sorbent will have an adsorption characteristic known as specific product which corresponds to the plant operating and design conditions. The specific product is defined as the quantity of gas produced in terms of normal cubic meters per hour per cubic meter of molecular sieve installed in the plant. The unit for specific product is therefore $m_n^3/hr.m^3$. The plant specific product may fluctuate in practice, depending on the operational mode of the plant to give optimum use of the molecular sieves. However, the design specific product will determine the amount of sieve required and hence air to plant flow rates. Vacuum pump flow rates may then be determined as a result of a mass balance of the air to plant flow rate and the product off take. The amount of sieve required will determine the size of vessels to hold the sieve. The shape of these should be economically designed to ensure a minimum bed height, which will result in a low pressure drop over the bed and therefore a lower plant power requirement.

The Author obtained a provisional process design package from BOC through Afrox Limited. This package contains the theoretical process design requirements on which a full pilot plant design was still required. The package contained the relevant information of air to plant flow rates, vessel, valve and pipework sizes as well as vacuum pump throughput. Many modifications to this package had to be made in terms of control and instrumentation. No such plant has yet been built to this design and the pilot plant will also serve to verify the BOC design.

The units of measurement in this section refer all process flows in terms of Normal cubic meters or m_n^3. This is the equivalent volumetric flow at a temperature of 273 Kelvin and at a pressure of 101,3 kPa absolute (Airco, 1988). This will always give an accurate value on which mass flow can be calculated. This figure also allows for the correct flow rates to be maintained in conditions where the gas density changes whenever the temperature or pressure changes. Actual cubic meters, or Am^3, represents actual operating conditions or actual process parameters. These actual figures are not very useful for mass balance calculations until they are converted to normal conditions by using Charle's Gas laws.

The BOC process plant design is based on the isotherms of a molecular sieve provided by UOP. This is called a UOP VSA II sieve. The sieve characteristics of this molecular sieve were shown in Table 3.1 and the isotherms shown in Figure 3.3. These sieve characteristics form the heart of the process design. The sieve volume is based on the selected molecular sieve specific product and it is determined by the following simple calculation:

Required oxygen flow rate = 313 m_n^3/hr
Specific product = 13,4 m_n^3/hr.m^3

Therefore, sieve required = Flow rate / specific product
= 313 / 13,4
= 23,36 m^3

Therefore, sieve volume per vessel = Total sieve volume / Number of vessels
= 23,36 / 3
= 7,78 m^3

This volume of molecular sieve will then be filled into each adsorber vessel.

A packing density of 670 kg/m^3 is expected using a constant density filling chute. This means that the mass of molecular sieve per vessel can be calculated.

Mass of sieve = Sieve volume * packing density
= 7,78 * 670
= 5220 kg

From the isotherms on Figure 3.3, it can be seen that the plant must operate between 1350 mbar (abs.) and 270 mbar (abs.) for efficient adsorption and regeneration of sieve. This affects the selection of machinery as the selected machinery must perform well when operating within these design pressure limits.

The plant design output is listed in Table 3.2. These figures are dependent on mechanical design, machine selection, sieve performance and product oxygen requirements. The specific power is calculated by BOC on a sophisticated computer package, however this is very dependent on the selection of machinery and is based on coastal atmospheric conditions in the United Kingdom with a pressure of 1013 mbar (abs.) and a temperature of 15°C.

DESCRIPTION	UNIT	RESULT
Flow	m_n^3/hr	313
Purity	% Oxygen	93
Temperature	°C	25
Specific Power	kWhr/m_n^3	0,45
Pressure	mbar (abs.)	1250

TABLE 3.2

EXPECTED PILOT PLANT RESULTS (BOC, 1988)

During the vessel filling procedure discussed in section 4.3.1, it was found that the desired mass of molecular sieve was not loaded into the adsorber vessels. After the vessels were vibrated and topped up with molecular sieve, the sieve mass was still not correct and this indicated that the correct packing density had not been obtained. The actual packing density can be calculated as :

$$\begin{aligned}\text{Packing density} &= \text{actual mass of sieve / vessel volume}\\ &= 4863\ /\ 7{,}78\\ &= 625\ \text{kg/m}^3\end{aligned}$$

This is significantly lower than the expected packing density of 670 kg/m^3. This lower density may have an affect on the plant performance. Without the aid of sophisticated computer design packages, it is difficult to accurately calculate any real consequences. As the plant specific product, the product recovery and the product flow rate are all closely related, a trade off between these parameters may be expected and these may be adjusted by the purge, backfill and equalisation flow rates to produce the optimum plant outputs.

The plant product recovery is defined as the amount of oxygen in the product stream as a percentage of the amount of oxygen in the feed stream. This can be calculated either on a mass basis or on a volume basis and in both cases they should produce the same result. The expected product recovery for the pilot plant can be calculated from the design air to plant flow rate and the design oxygen product flow rate. The oxygen content in both streams must also be measured in order to calculate the actual amount of oxygen contained in the relevant streams. The expected product recovery for the pilot plant is calculated on a volumetric basis as follows :

Product recovery = Oxygen out / Oxygen in
= 313 * 0,93 / 2690 * 0,209
= 51,8%

The actual product recovery obtained by the pilot plant is calculated as shown above and may fluctuate in the actual case due to variances in the plant operating parameters.

A layer of Rhone Poulenc type 5A activated alumina is inserted into the adsorber vessels to dry the feed air before it contacts the VSA II molecular sieve. Alumina drying systems have been used in air separation systems for many years to prevent moisture from entering cryogenic heat exchangers. The activated alumina is regenerated by the process vacuum pump during the desorption and evacuation stages.

3.3.1.1 Process Description

The three bed pilot plant process flow diagram is shown in Figure 3.8 and the ideal bed pressure profile is shown in Figure 3.9. A complete plant piping and instruments diagram (P&ID) is shown in Appendix 2.

The three adsorber beds 'A', 'B' and 'C' are operated 120° out of phase with each other in order to maximise the continuity of the product flow and pressure.

One complete three bed cycle is of approximately 180 seconds duration. Over this cycle period, each of the three adsorber beds is subjected to three process stages.

Stage 1) Production
Stage 2) Depressurisation
Stage 3) Purge / Pressurisation

Each stage is of approximately 60 seconds duration. Throughout each step process air is drawn into the process air blower through the air inlet filter and the blower maintains the feed air header at approximately 1500 mbar (abs.). The process air blower package ensures that the feed air entering the adsorber bed is cooled to the ambient temperature in an aftercooler and that large water droplets are removed in a condensate separator. Each adsorber vessel contains a bottom bed layer of activated alumina to remove any remaining moisture before the process air reaches the zeolite molecular sieve adsorbent which forms the majority of the packed bed.

The complete three bed cycle is described by assuming that bed A is commencing stage one of the process and should be read in conjunction with the flow diagram shown in Figure 3.8.

Saturated feed air is diverted to bed A via pneumatically operated valve XV1A. The bed is pressurised by the air blower to a pressure of 1350 mbar (abs.) and this pressure is maintained whilst nitrogen is adsorbed by the zeolite. This process causes the oxygen concentration in the product gas stream to increase so that the product removed from the top of the bed is at a purity of greater than 93% oxygen with the balance being a mixture of nitrogen and argon. Some of this gas enters the product buffer vessel whilst the remainder is passed into bed B via actuated valve XV3B to partially pressurise it in a backfill step. The oxygen enriched gas is passed from the product buffer vessel to the final oxygen application through the product valve XV81.

After approximately 60 seconds, adsorber bed A becomes saturated with nitrogen, but before any breakthrough of air can occur, the oxygen enriched product stream is diverted to adsorber bed B via actuated valve XV5B in an equalisation step. Bed B is at this stage regenerated and partially pressurised. The pressures of bed A and bed B start to average through the equalisation line at which point feed air is diverted from bed A to bed B via actuated valve XV1B. The equalisation valve XV5B linking adsorber beds A and B closes before cross over of pressure can occur and bed B is then fully pressurised by the air blower via actuated valve XV1B to approximately 1350 mbar (abs.) to supply product to the buffer vessel via actuated valve XV6B.

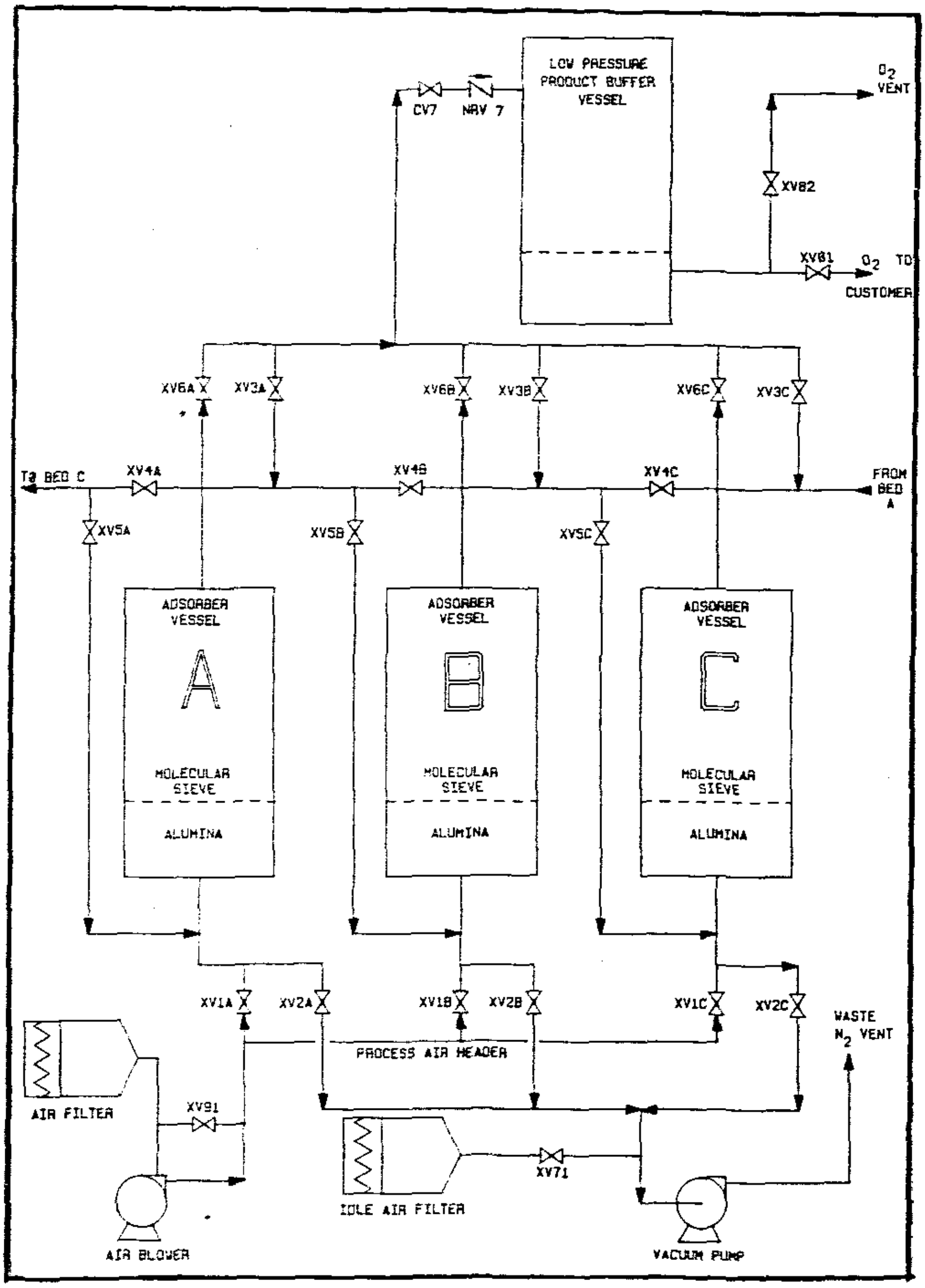


FIGURE 3.8

THREE BED VSA PROCESS FLOW DIAGRAM (BOC 1988)

Adsorber bed A then enters the process stage two of the cycle, the depressurisation and evacuation to regenerate the molecular sieve. The regeneration commences with the controlled depressurisation of the adsorber beds. This is achieved at the product end of the adsorber vessel with the depressurising gas from adsorber bed A being used as a purge stream for the regeneration of adsorber bed C via actuated valve XV4A. When the pressure in adsorber bed A has reached approximately 1050 mbar (abs.) actuated valve XV4A closes and adsorber bed A is evacuated with the vacuum pump to a final pressure of 270 mbar (abs.) via actuated valve XV2A.

At this point adsorber bed A enters the process stage three of the cycle, being the purge and backfill steps. A purge flow is introduced into bed A, countercurrent to the process air flow via actuated valve XV4B and the bed is purged whilst being held under constant vacuum, until the purge flow is terminated by the closure of actuated valve XV4B. The bed is then partially pressurised to approximately 800 mbar (abs.) with product quality gas through actuated valve XV3A in a backfill step. The vessel is then repressurised to atmospheric air pressure by equalisation with the most recently saturated adsorber bed, being bed C.

Adsorber bed A is now ready to re-commence process stage one and in this way a continuous supply of product quality gas is sent to the product buffer vessel and from there on to the final oxygen application.

The sequencing of the three process stages is achieved by pneumatically actuated valves which are automatically controlled by a programmable logic controller (PLC). The duration of each process stage is determined by the time to evacuate the regenerating adsorber bed down to approximately 270 mbar (abs.).

The pressure swing in the adsorber vessels during the normal operating cycle is between 1350 mbar (abs.) and 270 mbar (abs.) as shown on the pressure profile in Figure 3.9. The change of pressure during the VSA plant cycle can clearly be seen on a time scale. From the start of the cycle until 20 seconds, the vessel is at 1350 mbar (abs.) and is on producing oxygen as in stage one. The vessel then provides backfill to another vessel which reduces its pressure level to 1200 mbar (abs.). At 30 seconds from the start of the cycle, the vessel provides purge to the evacuated vessel and this reduces the pressure to approximately 1000 mbar (abs.). The evacuation step with the vacuum pump then begins until the pressure has reached 270 mbar (abs.) at 100 seconds from the start of the cycle. The vessel then receives purge which increases the pressure until 120 seconds. The vessel then receives backfill until the pressure has increased to 800 mbar (abs.). The vessel is then equalised to 1000 mbar (abs.). The air feed from the air blower is then admitted and the vessel is pressurised to begin production at 180 seconds from the start of the previous cycle.

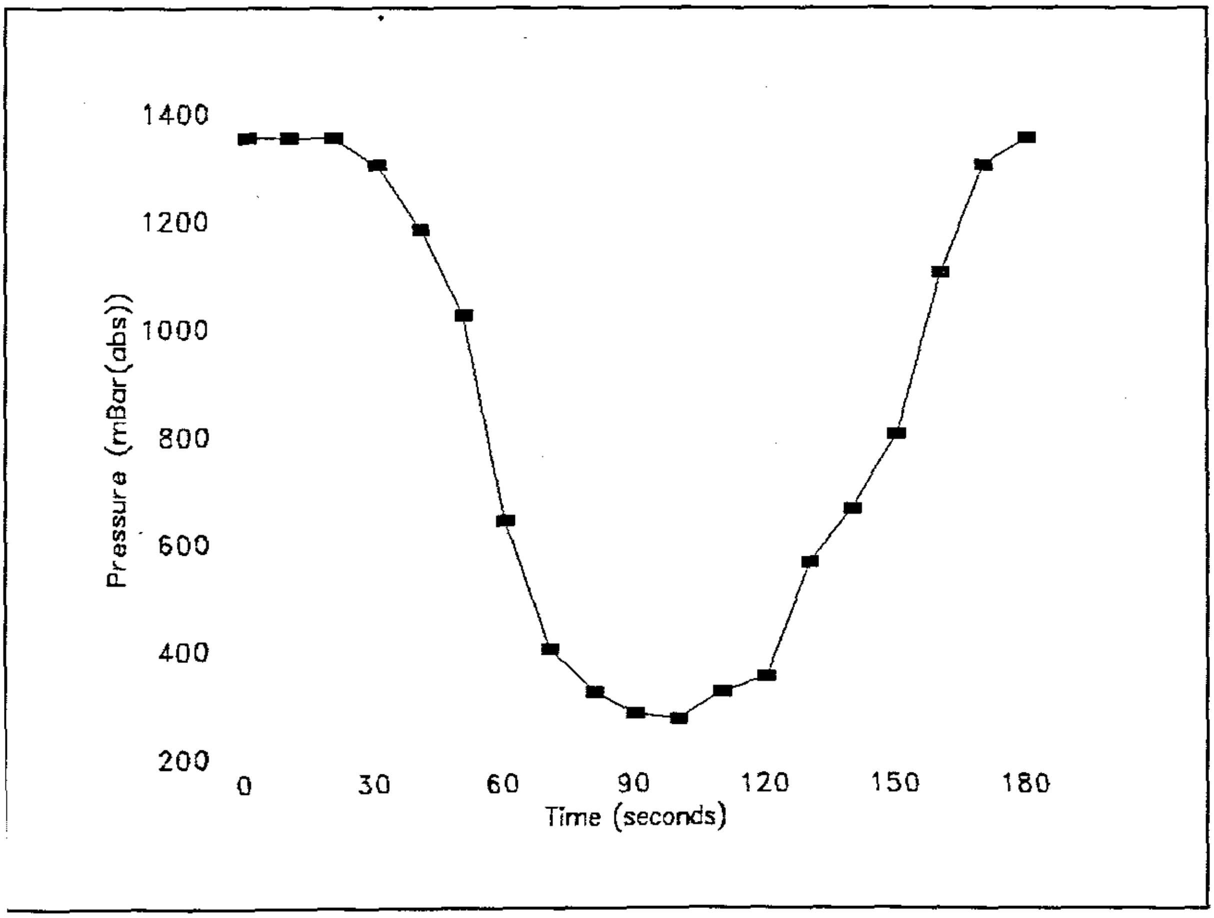


FIGURE 3.9

IDEAL BED PRESSURE PROFILE

The symmetry of the three adsorber vessels must be considered. There may be variations in the vessel volumes due to the manufacture and associated interconnecting pipework. This difference in volume may cause a difference in the pressure profile of each particular bed. This can be adjusted by controlling the gas flow during the purge, backfill or equalisation steps. It is critical that all three adsorber vessels follow similar pressure profiles to ensure continuation of production of product quality gas at the required purity, pressure and flow rate.

3.3.2 Mechanical Design

The choice of mainline equipment has a severe effect on the power requirement and operating costs of a PSA plant. Careful machine selection will therefore result in very low specific powers and installed plant costs. The mechanical layout and the integrity of the total plant construction will assist with the maintenance requirements and decrease the plant operational costs.

3.3.2.1 Pressure Equipment

The requirements for the air to plant flow were defined as (BOC, V1000, 1988) :

Temperature = 25°C
Pressure = 1000-1500 mbar (abs.) over a 1 minute cycle
Flow = 2690 m_n^3/hr (m_n^3 = 1013 mbar (abs.), 0°C)
Fluid = Dry atmospheric air.

To select and to correctly size the machine, the atmospheric conditions at the machine inlet must be considered. In the PWV area, the operating conditions (ambient design conditions) are as follows:

Temperature = 25°C
Pressure = 860 mbar (abs.)
Relative Humidity (RH) = 80%

It is necessary to compensate for moisture in the feed air stream by using steam tables and Dalton's law of partial pressures:

Relative Humidity, (RH), is defined as P_s/P_g;

where P_s = Vapour pressure,
and P_g = saturation pressure at 25°C,
and P_g = 3,166 kPa (Steam Tables).

Therefore, at the design ambient conditions of 25°C and 80% RH :

$$\begin{aligned} P_s &= RH * P_g \\ &= 0{,}8 * 3{,}166 \\ &= 2{,}5328 \text{ kPa} \\ &= 25{,}3 \text{ mbar} \end{aligned}$$

Dalton's law of partial pressure gives (Eastop & McKonkey, 1986 : 518) :

$$\begin{aligned} P_T &= P_A + P_S \\ 860 &= P_A + 25{,}3 \\ P_A &= 835 \text{ mbar (abs.)} \end{aligned}$$

Therefore, the affect of relative humidity is approximately 3% which is negligible.

To calculate the required dry air flow rate, we must consider the conditions at the inlet to the machine, (the subscript A denotes actual conditions, ie., the conditions that the machine will experience.)

At Inlet : P_A = 835 mbar
T_A = 25°C
Flow = 2690 m_n^3/hr

To compensate for normal flow conditions to actual flow conditions, Charle's Gas Law must be applied for unit flow:

$$\frac{P_A V_A}{T_A} = \frac{P_N V_N}{T_N}$$

$$\frac{835 * V_A}{298} = \frac{1013 * 2690}{273}$$

$$V_A = 3562 \text{ Am}^3/\text{hr}$$

$$= 59{,}4 \text{ Am}^3/\text{minute}$$

Therefore, it is essential to size a positive displacement machine to give a Free Air Delivery (F.A.D.) of 59,4 Am^3/minute.

There are various types of machinery that could provide the required air flow. The choice would depend on the required air discharge pressure to suit the choice of molecular sieve. The two most common machinery types are the Roots type and the single stage centrifugal type where a low pressure air feed is required. A sliding vane machine would be excluded, as at all times, oil free air must be provided.

The Roots type machine is of a two lobe type. One of the rotors is connected to the driving mechanism and the other is gear driven from the first. The rotors rotate in phase and the cycloidal lobe profile gives correct mating to seal the delivery side from the inlet side. This sealing continues until delivery commences. There must be a small clearance between the lobes and between the casing and the lobes to eliminate wear. This clearance forms a leakage path for the air which increases rapidly as the pressure ratio increases. The amount of leakage will determine the efficiency of the Roots type machine.

The centrifugal compressor consists of an impeller with a series of curved radial vanes. Air is drawn in near the eye of impeller, (or the hub) and is whirled around by the impeller vanes, as the impeller rotates at a high speed. Work is done on the air and it leaves the impeller with high velocity and increased pressure. As the air leaves the impeller tip, it is passed through diffuser passages which convert most of this kinetic energy into pressure energy.

The Roots type machine is preferred due to its relative simplicity, lower operating costs, efficiency and availability. Roots blowers can supply oil free air because the gear lubrication is protected from reaching the compression chamber by using a system of oil scraper rings, gland packing and distance pieces on the rotor shaft. Tender documents were prepared and following a tender adjudication an Aerzen machine was selected. From the Aerzen performance curves a GMb.15.11 (Aerzen, 1982) was selected.

The power requirement for a Roots type machine is given by (Eastop & Mckonkey, 1986 : 307), where P_1 and P_2 represent the inlet and outlet pressures respectively and V is the compression chamber volume and V_s represents the swept volume per minute through the machine. This power requirement is calculated as follows:

Work done per cycle = $(P_2 - P_1)\ V$
Work done per rev = $4\ (P_2 - P_1)\ V$
Work done per minute = $(P_2 - P_1) * V_s$

Therefore, Roots Work = $(P_2 - P_1) * V_s$
= (150 - 86) x 59,4
= 3802 KJ/minute
= 63,4 kW

Roots type machines generally compress according to the isentropic (i.e. reversible adiabatic) process, with the ratio of specific heats (n) for air being:

$$
\begin{aligned}
n &= C_p / C_v \\
&= 1005 / 718 \\
&= 1{,}4.
\end{aligned}
$$

Therefore, isentropic work (Eastop & McKonkey, 1986 : 307) can be expressed as :

$$\frac{n}{n-1} P V_s \left[\left(\frac{P_2}{P_1} \right)^{\frac{n-1}{n}} - 1 \right]$$

and the isentropic work can be calculated as :

$$\frac{1,4}{1,4-1} * 86 * 59,4\ [(\frac{150}{86})^{\frac{1,4-1}{1,4}} -1]$$

giving isentropic work = 3080 KJ/minute

= 51,3 kW

Therefore, Roots efficiency = isentropic work / roots work

= 51,3 / 63,4

= 0,81

The typical mechanical efficiency of a Roots type machine including the transmission and the motor losses is between 75% and 80% (Aerzen, 1982).

Therefore, by making the use of the calculation for Roots efficiency of 0.81 and making an assumption of an average mechanical efficiency of 0,77, the blower power requirements at full load conditions can be calculated. The power required at full load conditions is a relationship between the isentropic work, the mechanical efficiency and the Roots efficiency and is given as:

Power = Isentropic Work / Roots efficiency / Mechanical efficiency

$$Pwr = 51,3 * \frac{1}{0,81} * \frac{1}{0,77}$$

$$\mathrm{Pwr} = 82,3\ \mathrm{kW}$$

Therefore, a 90 kW Siemens motor was selected to allow for the power requirements at the maximum load conditions.

The specific power was calculated using the Aerzen blower curves from the manufacturers catalogue for the selected GMb 15.11 unit. For the purpose of this calculation we must assume the pressure increase to be linear from 1000 mbar (abs.) to 1500 mbar (abs.), during the production and pressurisation steps. It is necessary to calculate the operating pressure differential across the air blower between the machine inlet pressure (P_1) and the machine outlet pressure (P_2) to determine the absorbed power at the beginning (Pwr_1) and at the end (Pwr_2) of the production and pressurisation steps.

At the beginning of this cycle, the operating pressure difference, i.e.

$$P_2 - P_1 = 1000 - 860 = 140 \text{ mbar},$$

and, from the blower performance curves, the absorbed power is given as :

$$Pwr_1 = 27 \text{ kW}.$$

At the end of the cycle, the operating pressure difference, i.e.

$$P_2 - P_1 = 1500 - 860 = 640 \text{ mbar},$$

and, from the blower performance curves, the absorbed power is given as :

$$Pwr_2 = 81 \text{ kW}.$$

Therefore the average power demand during one complete cycle can be calculated as follows :

$$Pwr = (Pwr_1 + Pwr_2) / 2 = (27 + 81) / 2 = 54 \text{ kW}.$$

The blower specific power is a measurement of the power used by the blower per normal cubic meter of contained oxygen product (Design output flow is 313 m_n^3/hr of product at 93% purity which gives 291 m_n^3/hr oxygen).

Therefore, the expected specific power (SpPwr) for the air blower can be calculated as follows :

$$SpPwr = \text{Total Power / Oxygen Flow} = 54 / 291 = 0{,}185 \text{ kWhr}/m_n^3.$$

A powerview meter was installed on the air blower motor to constantly monitor the blower power requirements. This will record on an ongoing basis the motor amps, volts, power, power factor and instantaneous maximum demand. The powerview meter also contains an integrator to sum the total number of kilowatt-hour units consumed.

Roots machines have several shortcomings. These are associated with pressure waves caused by the discharge from the displacement volume to the delivery space. This wave moves at the speed of sound and is reflected from the approaching lobe to the delivery space. These pressure oscillations set up unsteady conditions in the delivery space which vary considerably depending on machine loading. The actual torque and loading on the rotors fluctuate with high frequency. This fluctuation is transmitted to the drive and creates difficulty with noise and vibration.

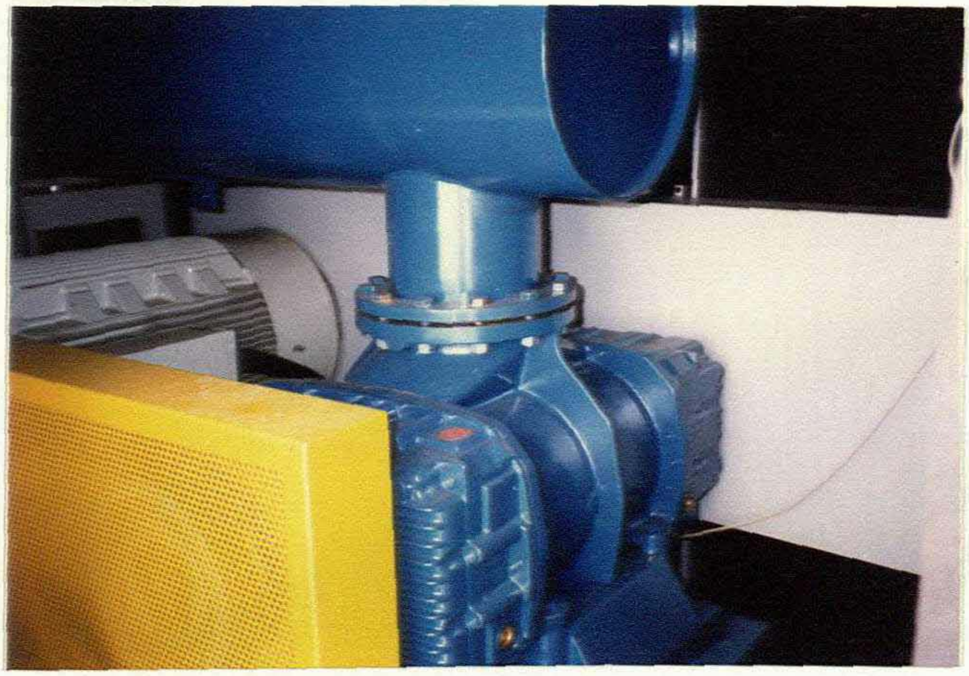


FIGURE 3.10
ROOTS BLOWER

In order to overcome these difficulties the selected Roots type blower was provided with four rubber anti-vibration mountings to minimise the vibration and an acoustic canopy was designed and installed to reduce the noise pressure levels to below 85 dB(A). The blower discharge pipework included a silencer and a resonator which assisted with the dampening of the pressure pulsations. The interconnecting pipework to the Roots blower package was connected with flexible pipe bellows to prevent fatigue failures due to the high vibrations expected in the process pipework. The selected Roots type blower is seen on the photograph as shown in Figure 3.10. In this Figure, the Aerzen Roots blower, Siemens motor, motor drive guards and the inlet silencer can all clearly be seen inside the acoustic canopy.

3.3.2.2 Vacuum Equipment

The requirements for the vacuum pump varied due to the cyclical nature of the process. The operating process design parameters were given as follows (BOC, V1000,'1988) :

Temperature = 25°C
Pressure = 900 - 270 mbar (abs.), varying over a 43 second cycle.
270 - 290 mbar (abs.) over 17 seconds. Then the pressure increases to 900 mbar (abs.).
Volume = Total volume to be evacuated in 43 seconds is 33,8 m_n^3. (m_n^3 = 1013 mbar (abs.), 0°C)
Fluid = Nitrogen enriched saturated air.

The vacuum pump flow is calculated from the sum of the vent flow volume (regeneration) and the purge flow. An allowance is added for pipe voidage and top bed voidage areas.

Vent flow	33,8 m_n^3
Voidage	1.2 m_n^3
Total	35 m_n^3

Therefore, a total equivalent volume of 35 m_n^3 is to be reduced in pressure from 900 mbar (abs.) to 270 mbar (abs.) in 43 seconds. This pumpdown is the most important part of the molecular sieve regeneration process.

To select and to correctly size the machine, the actual inlet flow at the beginning and end of each cycle must be considered and a machine must be selected to meet an average of these flow conditions. As the density of gas decreases proportionately with pressure, the gas to be evacuated will expand to constantly occupy the available space as the pressure is reduced. The total volume to be evacuated can be calculated at the operating pressure conditions at the start and the end of the evacuation cycle.

From Charle's Gas Law, the actual volume to be evacuated ($V2_1$) at the pump inlet conditions at the start of evacuation, is calculated as follows :

$$\frac{P_A V_A}{T_A} = \frac{P_N V_N}{T_N}$$

$$\frac{90 * V2_1}{298} = \frac{101{,}3 * 35}{273}$$

$$V2_1 = 43{,}0\ m^3$$

From Charle's Gas law, the actual volume to be evacuated ($V2_2$) at the pump inlet conditions at the end of evacuation, is calculated as follows :

$$\frac{P_A V_A}{T_A} = \frac{P_N V_N}{T_N}$$

$$\frac{27 * V2_2}{298} = \frac{101{,}3 * 35}{273}$$

$$V2_2 = 143{,}3\ m^3,$$

Therefore, the average actual volume ($V_{AVERAGE}$) to be evacuated in 43 seconds is calculated from an average of the actual inlet volumes at the start and end of the evacuation step.

$$\begin{aligned} V_{AVERAGE} &= (V2_1 + V2_2) / 2 \\ &= (43{,}0 + 143{,}3) / 2 \\ &= 93{,}2\ m^3. \end{aligned}$$

The average actual volumetric flow rate (V_{ACTUAL}) at inlet conditions is given by :

$$
\begin{aligned}
V_{ACTUAL} &= (V_{AVERAGE} / 43) * 60 \\
&= (93,2 / 43) * 60 \\
&= 130 \text{ Am}^3\text{/minute.}
\end{aligned}
$$

Therefore, it is essential to size the vacuum pump to give a positive displacement of 130 Am^3/minute.

There are various machines that could achieve such a flow rate and vacuum condition. The most common types are the Roots type, the sliding vane type and liquid ring machines.

The Roots type machine, as discussed in section 3.3.2.1 would usually be the most suitable. In order to achieve a low vacuum, two stages on a common drive shaft are connected in parallel, with water injection between the lobes would be necessary. The water injection would increase the pressure ratio by decreasing the clearance gap between the two lobes. The two stages will have to be used in parallel to achieve the volumetric flow rate at low inlet pressures in the required time. This is necessary to efficiently regenerate the molecular sieve. The shortcomings of the Roots type vacuum pump are as follows:

- the requirements for a water demineraliser for the water injection system;
- the separator to recover the demineralised water from the pump discharge;
- a large concrete foundation to support the heavy equipment;
- and the problems associated with high noise and vibration levels.

A sliding vane machine consists of a rotor mounted eccentrically in the machine body and supported by ball and roller bearings in the end covers of the machine body. The rotor is slotted to take the blades which are of a non metallic (usually asbestos) fibre. As each blade moves past the inlet port, suction takes place and then compression due to the decreasing volume between the rotor and the casing. Delivery begins with the arrival of each vane at the delivery port. Sliding vanes can achieve the required vacuum on a single machine and the correct pressure ratio would be obtained by adjusting the angles of the inlet and discharge ports with respect to the rotor location on the machine casing.

A liquid ring machine consists of a vane type impeller, which rotates in a liquid to compress gas trapped between the machine casing and the liquid ring. Due to the low VSA plant operating pressures the vapour pressure of the vacuum pump liquid could have an adverse effect on the power requirements. A large amount of energy is absorbed by heating and stirring this liquid. The volumetric efficiency is reduced by approximately 10% when using water as a medium due to the liquid vapour pressure that is occupying useful space. To achieve an ultra high vacuum, special vacuum oils may be used as a medium. For these reasons, a liquid ring machine is not altogether the most mechanically efficient type of machine for this process. A sketch showing the operation of the liquid ring type pump is shown in Figure 3.11.

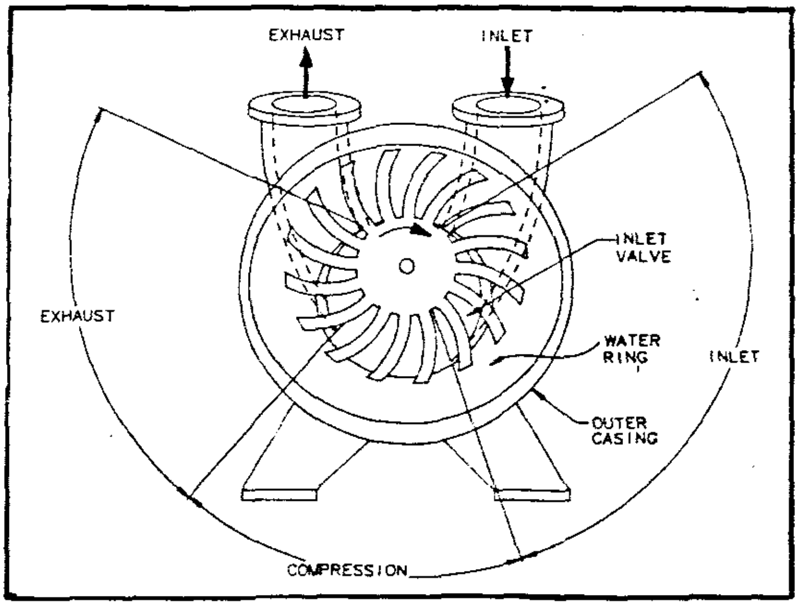


FIGURE 3.11

LIQUID RING VACUUM PUMP

Costs for all three options were obtained from Hibon, Siemens and Allis Chalmers for Roots, liquid ring and vane type pumps respectively, in order to conduct a fair comparison. The major differences between these machines are outlined in Table 3.3.

Item	Unit	Roots	Vane	Liquid ring
Manufacturer		Hibon	Allis Chalmers	Siemens
Capital Cost	R	505591	406131	248736
Power rating	kW	132	133	260
Noise levels	dB	110	95	70
Maintenance	R/Annum	15200	20300	2500

TABLE 3.3

COMPARISON OF VACUUM EQUIPMENT

The choice of vacuum equipment from the outline in Table 3.3 leads to quite a complex solution.

It was determined that the cost implications between the liquid ring machine and the sliding vane machine would be recovered within two years due to its lower power requirements. The Roots machine was unsuitable as it required a large concrete foundation to dampen the high vibration and this concrete foundation could not be skid mounted. On these assumptions, a sliding vane machine was selected. Later it was determined that in the event of a valve failure, a flow of oxygen enriched gas with a concentration of approximately 50% oxygen by volume could enter the inlet of the vacuum pump. This gas would cause a severe explosion should it reach the oil flooded vane casing. It was not possible to lubricate the sliding ferro-asbestos vanes with water and an oxygen compatible oil was prohibitively expensive. As a result, the liquid ring machine was then selected. The liquid ring machine could easily be skid mounted and has a short manufacture and delivery time of approximately eight weeks. Water carry over from the liquid ring pump would severely contaminate the VSA II molecular sieve. Therefore, a solenoid shut off valve on the water supply line, an automatic water drain valve, an automatic water shut off valve and a non-return valve were all fitted to the vacuum pump package to protect the molecular sieve from any possible moisture ingress.

From a liquid ring vacuum pump manufactures performance curves, a Torrvac

SV 11002 (Siemens, 1979) machine, operating at 442 r/min was selected.

The expected power figures for this particular liquid ring vacuum pump at the three relevant operating pressures were obtained from the vacuum pump performance curves at the corresponding operating pressure points, P_1 and P_2, which represent the vacuum pump inlet and discharge pressures respectively. The discharge pressure, P_2, remains constant as this is the atmospheric condition to which the vacuum pump discharges.

The power adsorbed at the start of the evacuation step is dependant on the operating pressure of the vacuum pump and is calculated as :

$$P_2 - P_1 = 860 - 900$$
$$= -40 \text{ mbar (abs.)}$$

The - sign implies that there is a positive suction pressure, and from the vacuum pump performance curves, the absorbed power, (Pwr_1), is given as:

$$Pwr_1 = 196 \text{ kW.}$$

The power adsorbed at the end of the evacuation step (i.e. the start of the purge step) is dependant on the operating pressures of the vacuum pump and is calculated as :

$$P_2 - P_1 = 860 - 270$$
$$= 590 \text{ mbar (abs.)}$$

and from the vacuum pump performance curves, the absorbed power, (Pwr_2), is given as:

$$Pwr_2 = 272 \text{ kW.}$$

The power adsorbed at the end of the purge step is dependant on the operating pressures of the vacuum pump and is calculated as :

$$P_2 - P_1 = 860 - 290$$
$$= 570 \text{ mbar (abs.)}$$

and from the vacuum pump performance curves, the absorbed power, (Pwr_3), is given as:

$$Pwr_3 = 240 \text{ kW.}$$

At the end of this purge step, the vacuum pump inlet pressure will change instantly to 900 mbar (abs.), where the cycle will begin again.

From the power curves a 315 kW GEC motor was selected, to allow for the power requirements at the maximum load condition.

Assuming a linear pumpdown in pressure and using the vacuum pump absorbed power curves from the manufacturers catalogue the expected average power over a complete cycle can be calculated.

The average power during evacuation (Pwr_{EVAC}) is given by :

$$\begin{aligned} Pwr_{EVAC} &= (Pwr_2 + Pwr_1) / 2 \\ &= (272 + 196) / 2 \\ &= 234 \text{ kW.} \end{aligned}$$

The average power during the purge step (Pwr_{PURGE})is given by :

$$\begin{aligned} Pwr_{PURGE} &= (Pwr_3 + Pwr_2) / 2 \\ &= (240 + 272) / 2 \\ &= 256 \text{ kW.} \end{aligned}$$

Therefore, the average power demand over one complete cycle can be calculated from the evacuation and purge powers as :

$$\begin{aligned} Pwr &= \{(Pwr_{EVAC} * 43) + (Pwr_{PURGE} * 17)\} / 60 \\ &= \{(234 * 43 + 256 * 17)\} / 60 \\ &= 240 \text{ kW.} \end{aligned}$$

Therefore, the expected vacuum pump specific power (SpPwr) can be calculated from the total power and the oxygen product flow rate as follows:

$$\begin{aligned} SpPwr &= \text{Total power / oxygen flow} \\ &= 240 / 291 \\ &= 0.825 \text{ kWhr/m}_n^3. \end{aligned}$$

This figure is significantly higher than the expected specific power of a Roots type machine recommended by BOC with a specific power of 0,285 kWhr/m_n^3. This will have a major impact on the operating power costs of the pilot plant as the selected liquid ring pump specific power is nearly three times larger than the recommended Roots type machine.

A Powerview meter was installed onto the vacuum pump motor to constantly monitor the vacuum pump power requirements. This will record on an ongoing basis the motor amps, volts, power, power factor and instantaneous maximum demand. The powerview meter has an integrator function to totalise the kilowatt-hour units consumed.

Liquid ring pumps have previously been applied to this type of process. The main shortcoming of this type of pump is that in certain cases the liquid ring may collapse as the inlet of the vacuum pump is diverted from one adsorber bed at the end of the purge step to the next adsorber bed at the beginning of the evacuation step and the pump inlet experiences an instantaneous change of pressure from 290 mbar (abs.) to 900 mbar (abs.). This 610 mbar pressure change may result in the liquid ring collapsing and a large volume of water being discharged into the sump. This could lead to system flooding or water carry over into the waste nitrogen vent. The selected vacuum pump assembly is shown in Figure 3.12.

FIGURE 3.12

VACUUM PUMP ASSEMBLY

In this Figure, the Torrvac (Siemens Elmo-F) vacuum pump can be seen mounted on to the equipment skid. The inlet water pipework painted green can clearly be seen. The inlet manifold conveying the waste nitrogen is situated above the machine on the left hand side and the discharge pipework seen on the right hand side is discharging the waste nitrogen and water mixture into the sump, situated underneath the vacuum pump. The waste nitrogen discharge vent and the GEC motor can be seen in the background.

3.3.2.3 Vessels and pipework

The VSA plant adsorber vessel volume is determined by the quantity of molecular sieve required for the VSA process. The vessels are designed to hold the molecular sieve with a minimum bed height, which will maintain the differential pressure drop across the bed to a minimum. The molecular sieve characteristics also dictate the relevant operating pressure ranges of the vessels and pipework and these items must then be designed accordingly.

The three vertical adsorber vessels were designed in accordance with Asme VIII div. 2. (BOC V1000, 1988) This design code is an internationally recognised code requiring very high standards, compared to the BS (British Standards) and DOT (American Department Of Transport) standards. These codes define the grades of carbon steel that may be used during construction and outline the vessel design, manufacturing and welding procedures. The ASME VIII div. 2 code was recommended by BOC and the design calculation was completed by utilising a computer package available at IRD, the selected vessel manufacturing company. The operating and design pressure ranges of the vessels were to incorporate a cyclical pressure loading between a full vacuum and 2000 mbar (abs.) and the design life was selected as 20 years. The vessels were built with a skirt and foot ring to give additional support to the vessel skid. The vessels were Post Weld Heat Treated (PWHT) at 600°C for one hour to passivate the welds and to relieve the stresses created during the rolling of the shell material.

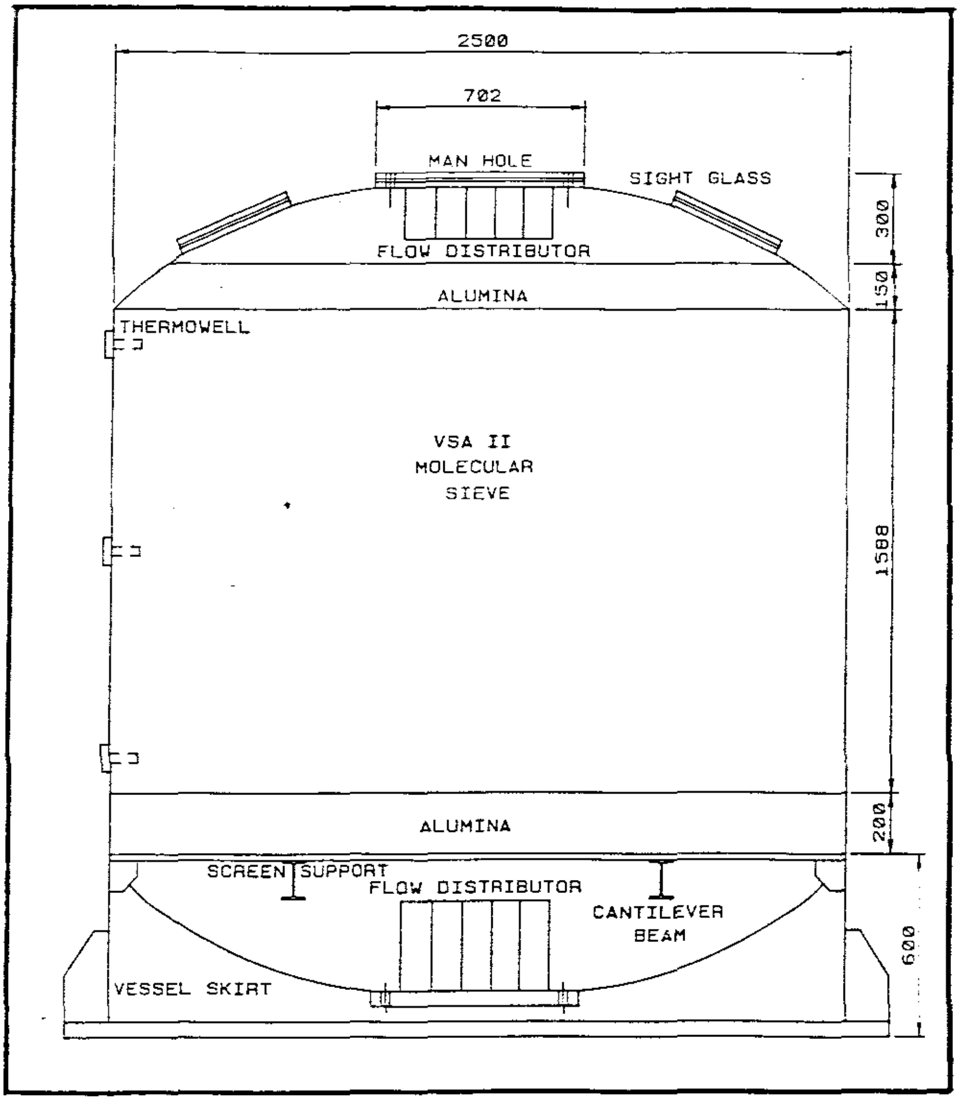


FIGURE 3.13
ADSORBER VESSEL DETAILS

The most critical design requirement is that the bed height must be kept to a minimum. The geometrical layout of the VSA plant adsorber vessels is shown in Figure 3.13. This critical bed height is specified in the design package as 1588 mm. This is done to decrease the pressure drop over the bed and therefore keep power requirements to a minimum. In addition to these requirements, various additions and modifications to the vessels were made.

The top and bottom voidage areas were kept to a minimum to ensure the maximum recovery of useful product. Flow distributors were manufactured from a stainless steel wedgewire screen and were installed at the top and bottom entrances to the vessels to eliminate the possibility of drift losses, channelling, to ensure that flow distribution takes place, and that turbulent flow is introduced into the vessels. Torrispherical dished ends were used on the adsorber vessels as they help to reduce the top bed voidage area because they occupy less contained area than ellipsoidal dished ends. Torrispherical dished ends are however more expensive than ellipsoidal dished ends.

The molecular sieve screen support was built to support the molecular sieve and to help provide further flow distribution. These supports were manufactured in the form of a grating from 50 mm flat bar, crossed with 22 mm round bar, all supported from the bottom dished end on a cantilever ring and two parallel 100 mm "I" beams. The total mass that the bed support was required to hold was approximately 6.7 metric tonnes. A 0,5 mm stainless steel wedgewire screen was placed on top of this grating, to provide further flow distribution and bed support. The edges were sealed with a stainless steel ring and an asbestos rope seal to prevent flow channelling up the vessel sides and to prevent migration of the molecular sieve particles.

Weld tags were located on the inside of the vessel to indicate the level to which the zeolite molecular sieve and activated alumina layers should be filled. Further additions were two sight glasses on each of the top dished ends, to allow for a visual inspection of the molecular sieve condition to be undertaken. This will ensure that top bed fluidisation (mixing of the two top bed layers) can easily be detected. A manhole was built into the top dished end to give access to the vessel interior for inspection and testing. The three different layers of molecular sieves were also installed through this manhole, by using a constant density filling device.

The vessel interior was shot blasted, degreased and left natural. Paint, galvanizing and zinc oxide spraying were not considered to prevent corrosion as these may peel and cause contamination of the molecular sieves. The moisture from the air is removed in the alumina layer and therefore any corrosion should be limited to the bottom dished end only.

The pipework was designed in accordance with ASME VIII Div. 2. This was due to the fact that there is a larger vacuum than pressure difference with respect to atmospheric conditions. The pipe material selected was ASTM A106 Grade B, schedule 40, seamless pipe. ANSI B31.3 welding procedures were selected as these had previously been used by the author in the construction of oxygen supply pipework at high pressure conditions. The pipe diameter was determined by the velocity of the gas in the pipes which was always sonic velocity at the operating conditions. These pipe diameters were recommended by BOC using computer design packages and these recommendations were fulfilled (BOC V1000, 1988). All pipe runs were internally pickled using a standard pickling procedure. Pickling pipework removes all of the oils and greases on the pipework that can be very hazardous in the presence of oxygen. These pipe runs were individually tested to the operating pressure and vacuum conditions, incorporating a large safety factor as schedule 40 pipe can withstand a pressure of up to 600 Bar.

Gasket material was to be oxygen compatible. Various oxygen compatible gaskets such as Teflon and compressed asbestos fibre (CAF) were tested on a test rig to ensure their compatibility to the vacuum swing cycle and these gaskets failed under the full vacuum conditions. A Viton gasket material was eventually selected and was found to be compatible with oxygen at the VSA plant operating pressures and temperatures.

A photograph showing the vessel skid assembly is shown in Figure 3.14. This Figure shows the vessel skid assembly was transported on a standard low bed trailer to the test site in Pretoria. Careful design optimisation was required to enable a plant capable of producing 10 TPD oxygen to be transported as an un-escorted abnormal load. This load did not require any Escom of Telcom permits as the height did not infringe any telephone cables or electricity pylons. The vacuum manifold can be seen under the vessels and the blanked off flange will be connected to the inlet of the vacuum pump package. The vessel skid assembly has a mass of 75 tonnes and a special jacking system had to be used for loading and unloading the skid assembly on to the standard low bed trailer which does not require the use of cranes. The three adsorber vessels and the interconnecting pipework on the vessel skid were all prefabricated in the workshops of Industrial Research and Development.

FIGURE 3.14
VESSEL SKID ASSEMBLY

3.3.3 Control and Instrumentation

The VSA pilot plant requires large amount of control and instrumentation to ensure smooth plant operation and that suitable measurements can be taken to record process operating characteristics. The control system ensures that the actual cycle follows the theoretical process cycle as closely as possible and should activate alarms and trip functions if the system malfunctions. The level of instrumentation added to a pilot plant is far in excess of the level that would normally be installed onto a commercial unit. The additional instrumentation was added to give more information for the understanding of the adsorption separation process.

3.3.3.1 PSA system control

Pressure

The adsorber bed pressure is determined by the process air pressure from the selected Roots blower, the product oxygen take off rate and the cycle time.

The VSA plant should be able to supply product up to the maximum pressure achievable by the selected Roots blower (1500 mbar (abs.) in the case of the pilot plant), less the pressure drop over the plant, caused by pipe friction and the bed differential pressure, which will usually allow a maximum product pressure of 1350 mbar (abs.).

The Roots blower and liquid ring vacuum pump packages have been separately provided with protection from high and low pressure conditions in the form of electronic pressure switches, safety valves, vacuum breakers, idle air inlets and recycle equipment.

The three adsorber bed pressures are plotted against time on a three pen chart recorder. This will give a visual indication of the current pressure status of the adsorber vessels. The pressure profiles can be adjusted by changing the purge, equalisation and backfill flow rates.

Should the instrument air system fail due to a low pressure, a signal will be sent to the Programmable Logic Controller (PLC) to put the VSA into a controlled shutdown sequence. This will allow for the VSA plant to equalise the bed pressures before shutting down, which prevents any potential damage that may be caused by top bed fluidisation on plant re-starting due to uneven bed pressures.

Flow rate

Product flow control is provided by means of manual throttle valves (butterfly type) combined with the process actuated on off valves. These manual throttle valves are located upstream from the process actuated valves. The actuated valves open fully to allow the flow to begin and the amount of flow is controlled by the opening of the manual throttle valve. This action will control the purge, equalisation and backfill flow rates and will have a corresponding influence on the bed pressure profile. Vacuum pump and Roots blower packages have automatic flow controllers and trips for process utility streams (i.e. high temperature, low pressure etc.). The specific flow rates as mentioned in the discussions on the Roots blower and vacuum pump packages in sections 3.3.2.1 and 3.3.2.2 will be closely monitored and controlled. Flow rate calculations are undertaken for the air to plant and the oxygen product flow rates in terms of Nm^3/hr.

Product Purity

The product purity is a function of the product flow rate and the final vacuum level (once the purge and backfill flow rates have been set during the plant commissioning). Control of purity is to be achieved by controlling the product flow rate or by adjusting the regenerating vacuum level between 180 mbar (abs.) to 270 mbar (abs.). The control of vacuum level can be obtained by adjusting the volume of purge flow through the manual throttle valve or by changing the evacuation step duration. Changing the step duration will affect the total time of the part cycle. The product purity should be 93% oxygen for the design flow rate. The product purity should be better than 93% for all flow rates below the design flow rate but never greater than 95% oxygen. The pilot plant PLC was programmed to constantly monitor the product purity and to sound an alarm if the purity should fall below 90% oxygen. The plant should also shut down if the purity falls below 85% oxygen. A manual purity over ride switch was added to allow the plant to operate below 85% oxygen product purity which was useful during the initial plant start up.

Temperature

The pilot plant does not incorporate any process temperature control, although product purity and final vacuum level will be influenced by ambient temperature conditions. There is a significant difference between design temperature and ambient conditions, therefore it is expected that the pilot plant product purity may decrease due to the difference in mass flow rates caused by the thermal expansion and contraction of gases during very hot or very cold ambient conditions. It is expected that cold spots may form on the vessel shell due to endothermic (heat absorbing) reactions between the molecular sieve, alumina and the waste nitrogen during the evacuation step.

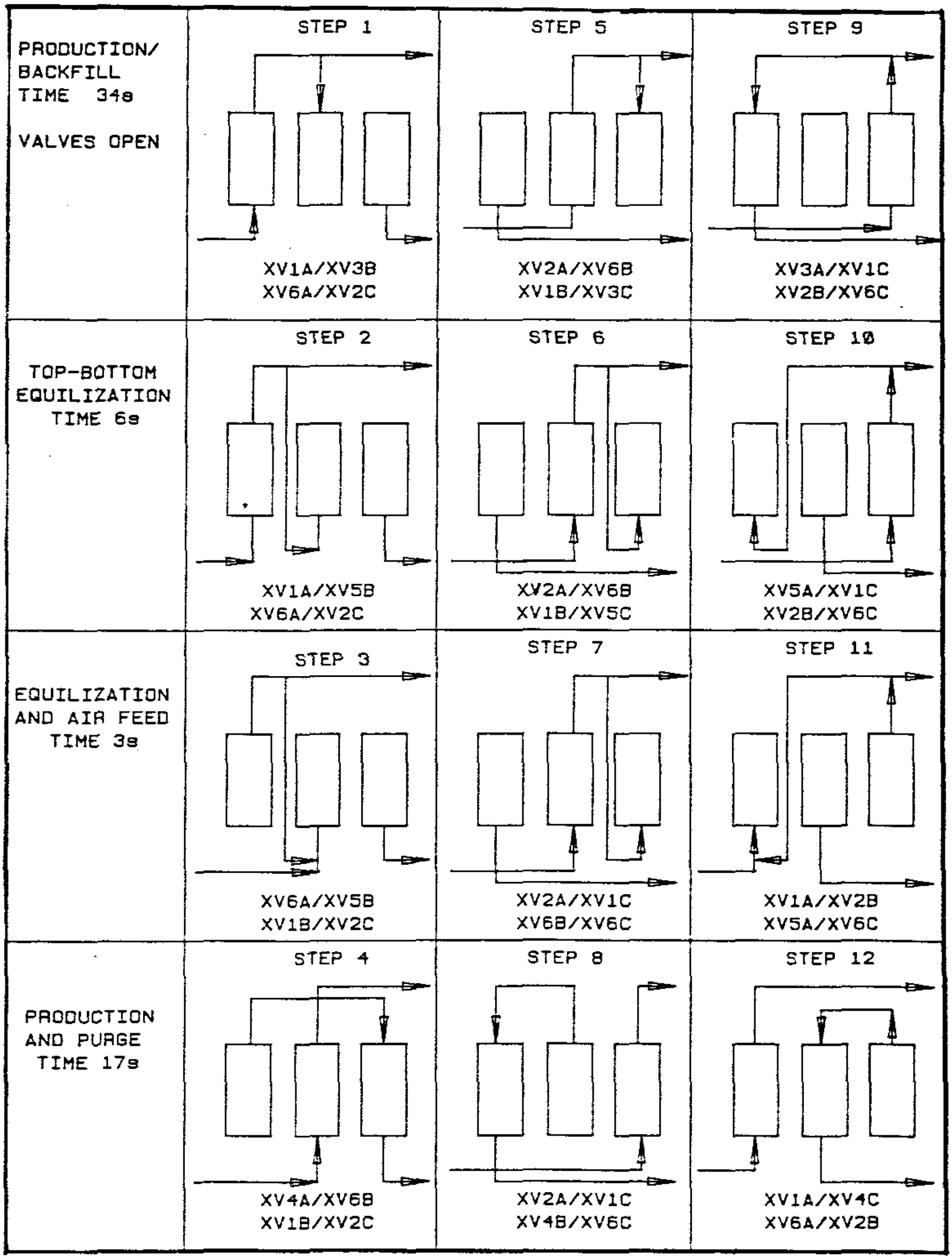


FIGURE 3.15

FULL THREE BED PART CYCLES

System Sequencing

The sequencing of the system is achieved by pneumatically operated high performance butterfly valves and actuators as supplied by Fisher and these are controlled and actuated by a programmable logic controller (PLC), as supplied by Texas Instruments. The PLC program is compiled to achieve the part cycles shown in Figure 3.15.

Valve overlaps have been added to prevent inter-bed gas flows and dead-ending of the mainline equipment. These overlaps are typically less than one second in duration and they can be set during the plant commissioning, by reprogramming of the PLC.

CYCLE	STEP	HIGH PRESSURE BED mbar (abs.)	LOW PRESSURE BED mbar (abs.)
Equalisation	1,5,9	1325	750 (650) Min
Equalisation (Feed)	3,7,11	1200	920 (850) Min
Production with Purge	4,8,12	1000 - 1350	270 - 330

TABLE 3.4

INTERSTAGE PRESSURE LIMITS

The normal operating cycle for the pilot plant is built up by three part cycles, each with a duration of 60 seconds. Each part cycle comprises four steps being, production with backfill, top to bottom equalisation, equalisation with air feed and production with purge. This leads to twelve part cycles for a complete adsorption and regeneration on one adsorber bed. The full cycle is shown in Figure 3.15 and this the valve openings shown should be read in conjunction with Figure 3.8. It is important that the operating pressures at the start and end of these steps are correct and they must be monitored at all times. The required pressure ranges before any part cycle can commence is shown in Table 3.4 as interstage pressure limits. These are also known as bed permissives as the PLC will not allow the cycle to continue unless these conditions have been met. These pressures will also be monitored by the PLC and will cause the plant to undertake an emergency shut down should a faulty process condition occur. This emergency shut down procedure is important as high flow velocities entering the adsorber vessels at incorrect pressures could cause the alumina, molecular sieve and ceramic balls to mix. This phenomenon is called bed fluidisation and these molecular sieves would then have to be replaced at a large financial expense.

3.3.3.2 Instrumentation

In order to control the VSA plant and to give meaningful indications, various instruments were added in addition to those recommended by BOC. The main instruments to record data required for commissioning, performances evaluation and mass flow calculations (MFC) are outlined in Table 3.5.

Stream	Type	Range	Comment
Air to plant:			
Flow rate	Annubar	2500 - 3000 m_n^3/hr	Fully P-T compensated.
Pressure	PT	900 - 2000 mbar (abs.)	To PLC for MFC.
Temperature	RTD	-20 - 100 °C	To PLC for MFC.
Product Oxygen:			
Flow rate	Annubar	200 - 500 m_n^3/hr	Fully P-T compensated.
Pressure	PT	900 - 2000 mbar (abs.)	To PLC for MFC.
Temperature	RTD	0 - 100 °C	To PLC for MFC.
Purity	Servomex	90 - 100 % O_2	Fully P-T compensated.

TABLE 3.5

FLOW INSTRUMENTATION

In addition to the outline in Table 3.5, the following instrumentation was also added to the pilot plant :

Oxygen purity	:	A sample tube is connected to the top of each adsorber bed to give the product analysis from each bed (for commissioning purposes only). The oxygen purity measured at the product buffer vessel outlet was plotted against the product flow rate on a separate chart recorder.
Bed Pressure	:	An absolute pressure transmitter (PT) is installed at the top of each adsorber vessel and is

connected to a chart recorder to plot the pressure profiles of the three beds.

Bed Temperature : Resistance Temperature Devices (RTD) were installed to plot the bed temperature profiles and to monitor any cold spots that may occur.

Flow Rates : The air to plant flow rate and oxygen product flow rate were plotted against each other on a chart recorder. The mass flow rate calculations are pressure and temperature compensated to give a display and chart printout in terms of m_n^3/hr and to integrate the total flow rates in terms of kg/hr.

Instrument Selection

The following instruments were carefully selected to give the correct measurement tolerances, calibration ranges and the outputs that were mentioned above.

Flow Annubar : An annubar is a low pressure flow meter suitable for the operating pressure ranges of the air to plant and the product oxygen flow rates.

Temperature RTD : A temperature RTD is far more accurate than a thermocouple, however, as they have a slower response time a narrow 4,5 mm tube was selected.

Pressure Transmitters: Smar absolute pressure transmitters were selected due to their ability to update information instantaneously to give an accurate recording of the bed pressure profiles.

Chart recorder : A fast chart speed of 12 metres per hour is required during commissioning to give a clear indication of the rapidly changing bed pressure profiles. Chino three pen chart recorders with a digital display and variable speed drive were selected.

Analysis : A Servomex series 1100 with auto calibration, pressure and temperature compensation gives very reliable information and was selected.

PLC : A Texas Instruments model 435 PLC that can undertake complex flow calculations was selected and the TI-SOFT software packages are readily available for the ease of PLC programming.

Process Valves

The process actuated valves are represented as the items marked XV in Figure 3.8 and they had to be high performance butterfly type valves. It was estimated that these valves would be required to perform one million cycles in 9 months of continuous operation. These ranged between 300 mm diameter (vacuum valves) to 75 mm (purge valves).

Various valve types were considered. Few manufacturers were willing to guarantee their valves to one million cycles. Finally, stainless steel Fisher valves with viton seats were selected. These were supplied with Fisher double acting pneumatic actuators and were manufactured to conform with high internal specialist manufacturing standards used by Fisher (Fisher, 1991) in the USA. The supplied valves were totally degreased and cleaned for oxygen service to prevent any possible explosion caused by an oil and oxygen mixture.

The control and instrumentation mentioned in this section forms the basis of the instrumentation hardware and software selected for the construction of the VSA pilot plant. It is also possible to monitor and control these operating conditions by utilising alternative hardware and software and it was not possible to examine all of these alternatives. The selection was based mainly upon local supply, availability and familiarity with certain types of instrumentation equipment.

4 EVALUATION AND RESULTS

4.1 GENERAL

This chapter contains all the relevant tables, charts and graphs obtained from information collected to evaluate the different PSA plant layouts as outlined in section 2.3. The various PSA plant layouts are evaluated according to their commercial factors and different PSA suppliers are briefly discussed. The pilot plant construction and performance testing is discussed in detail. Where a commercial evaluation has been undertaken, all figures are related to operating conditions in the Pretoria, Witwatersrand, Vereeniging (PWV) area and wherever possible, locally available equipment has been selected.

4.2 VIABILITY OF PSA TECHNOLOGY

PSA plants can be evaluated according to capital costs, power costs and total plant operating costs. Where different process plant outputs are concerned, these costs are approximated, usually according to the rule of 0,6. This is a rule commonly used to scale costs of chemical process plants and is given by (Peters and Timmerhaus, 1988 : 166) as :

$$\text{cost x} = \text{cost y (size x/size y)}^{0.6},$$

where x and y represent the respective costs and capacities of different plant layouts. This indicates the cost implications as plant capacity increases and process cycles remain the same.

Capital costs for these plants typically **include** the following items :

- Pressure vessels and associated pipework;
- Air compressor or Roots type blower;
- Vacuum equipment (if required);
- Process valves;
- Mechanical installation;

- Electrical installation and distribution;
- Civil works;
- Molecular sieves;
- Instrumentation and process valves;
- Services (i.e. cooling towers, etc.);
- Project management fees;
- Commissioning and training;
- Contingency and escalation.

Capital costs for these plants typically **exclude** the following items :

- Oxygen compression systems;
- Telemetry facilities;
- Small items, eg.: Fencing, roadways, security, safety equipment, telephones, etc.;
- Liquid oxygen back up system.

The plant power costs are determined by examining the various Escom and municipal tariff rates in the PWV area. These tariff rates vary quite considerably and have a major influence on the maximum demand costs. The cost per kWhr remains fairly constant and these amounts were averaged out at a R 0,07 per kilowatt hour (1992). This average electricity unit charge has been used to compare the various power costs of oxygen PSA plants. The electricity cost has been included in the plant costing that has been shown as a Rand per tonne figure.

Plant operating costs are derived by incorporating the power costs and depreciating the equipment on a straight line basis over a ten year period. This assumes a ten year contract period and at the end of the contract period the capital equipment will have a residual value exceeding the original amount borrowed to purchase the equipment. This cost includes an 18% interest rate on capital equipment. Also included is an allowance of 3% of capital cost for annual spare parts requirements (Coulson, Richardson, Sinnot, 1986 : 196). Operating overheads, including the costs for a site manager, maintenance fitter, electrician and instrumentation technician were estimated from known costs of typical cryogenic air separation units. These are included into the costs for both three bed VSA plants and for two bed PSA plants. These plant costs are reduced to a Rand per ton cost that would be faced by the oxygen user.

4.2.1 Pressure atmospheric plants

In determining the costs of pressure atmospheric plants, the costs for various sized units were obtained from Alpha Air Separation, local agents for an American PSA manufacturer, Airsep and from BOC. Various other plant sizes were estimated during 1992 and 1993 for different applications and these have been included into this graph to complete the cost and curves to give usable figures.

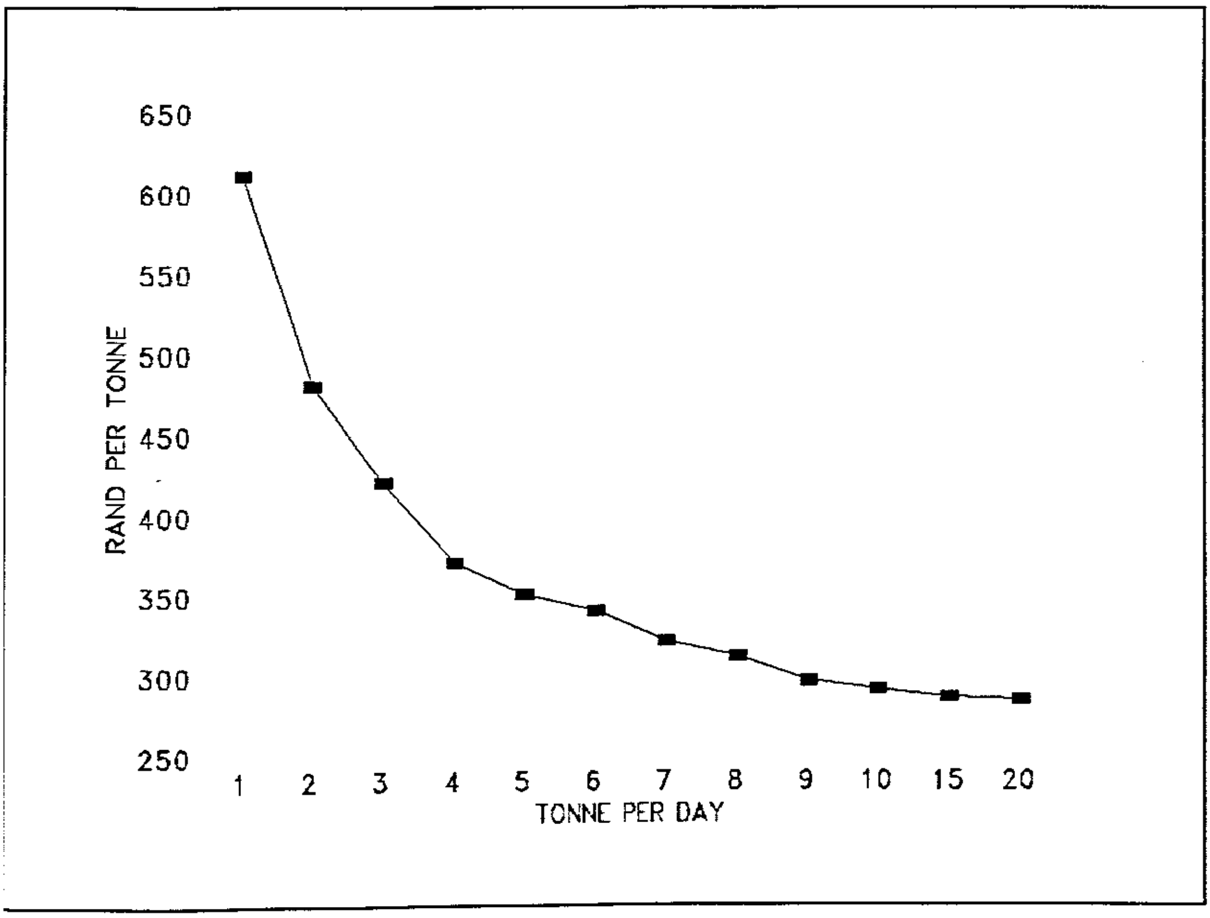


FIGURE 4.1

COST CURVE: TWO BED OXYGEN PSA

A graph has been projected on Figure 4.1 to show the Rand value per ton of oxygen that would be faced by the customer for an economic range of supply from a two bed oxygen PSA. This cost includes the mainline equipment, the power costs incorporating kiloWatt-hour units and expected plant maximum demand (taking the plant specific power requirements to lie between 0.7 and 0.9 kWhr/m_n^3) and plant operating overhead costs. At this stage product oxygen compression has not been included as the cost of these systems can greatly influence the entire plant capital and operating costs.

From the curve shown on Figure 4.1 it can be seen that the costs of the two bed oxygen PSA plants follows an exponential curve. This implies that as the plant output capacity increases the unit cost (in this case per tonne of oxygen) will decrease. The graph tails off at approximately 20 TPD as this is where these plants become uneconomical. For a supply in excess of 20 TPD, a system of more than one train of two bed PSA plants may sometimes be installed, having a similar cost implication to the end user as shown on the cost curve at the reference point of one of the two installed plants.

4.2.2 Pressure vacuum plants

During the course of the project lifespan, it was necessary to accurately estimate the installation costs of several large VSA plants in the PWV area. Estimates were conducted on these plants using outline process information obtained from both BOC and Showa Engineering. The plant costing was based on actual conditions wherever possible. The graph shown in Figure 4.2 is completed by using scale factors to give a complete cost curve.

This Figure shows the Rand per tonne value that would be faced by the customer for oxygen generated by a large three bed oxygen VSA plant. This cost includes the capital value of the mainline equipment, the power costs including both kilowatt-hour units and plant maximum demand (assuming that the plant specific power lies between 0.45 and 0.5 kWhr/m_n^3) and the plant operating overhead costs. At this stage of the evaluation oxygen product compression has not been included as the costs of these systems can greatly influence the plant capital and operating costs.

From the shape of the curve in Figure 4.2 it can clearly be seen that the costs of the three bed oxygen VSA plants are economical for a range of plant capacities from 15 TPD to approximately 100 TPD. A large step change occurs at approximately 100 TPD due to the specialised manufacturing techniques of pipework and molecular sieve handling that were discussed in section 2.3.2.3. The most economical plant options appear to be approximately between 50 TPD and 70 TPD as at this point the slope of the graph is at a minimum and this implies that the unit cost for oxygen is unlikely to decrease any further.

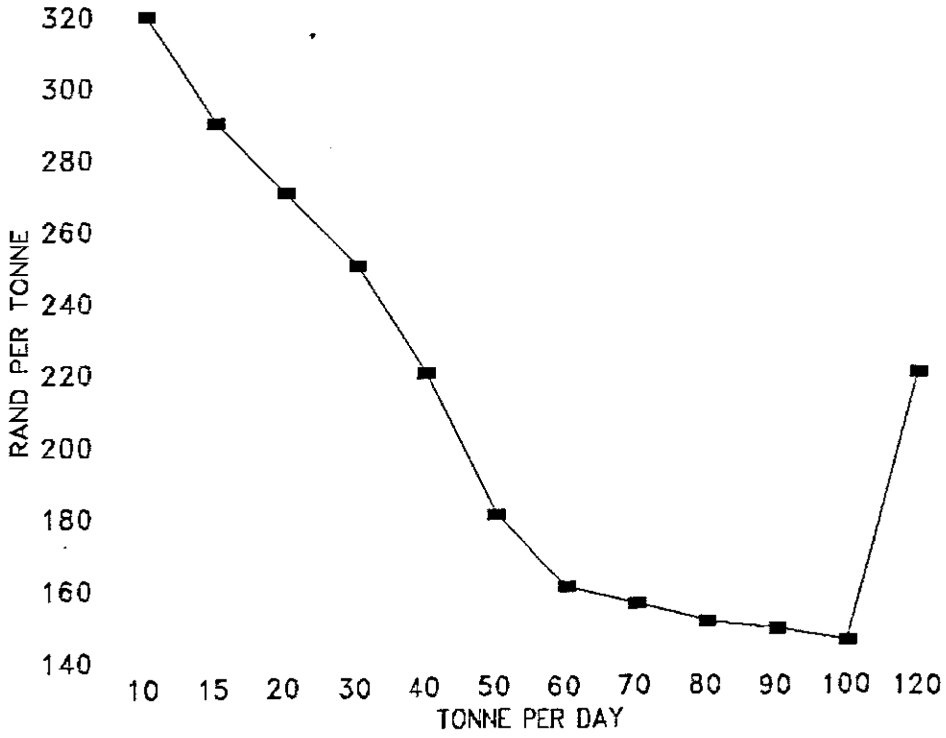


FIGURE 4.2

COST CURVE: THREE BED OXYGEN VSA

.2.3 Japanese Technology

Following a visit to Japan, the available Japanese PSA technology was thoroughly examined. The advantages and disadvantages are listed in Table 4.1 and an analysis of Japanese suppliers is shown in Table 4.2.

ITEM	ADVANTAGE	DISADVANTAGE
Research	More engineers and scientists working since the early 1970's have produced world leading results.	
Technology	Japanese are world leaders.	May give outdated technology to South Africa.
Power	Japan is power sensitive, lowest power consumption.	Low power costs mean high capital costs. This would not suit South African economy.
Trade	"New South Africa" policies allowing free trade with Japan.	Language barrier will be a problem. We will always have to work through an agent.
Mainline Equipment	Japanese equipment usually cheaper than European.	Spare part availability, maintenance and understanding will present many more problems.
Manufacturers	Many manufacturers will stimulate competition.	Manufacturers may deal directly with South African customers and this will prevent technology control.
Support		Japanese may not give required project support.
Cost		Weak Rand value, high import duties and freight will increase plant costs.

TABLE 4.1

JAPANESE TECHNOLOGY: ADVANTAGES AND DISADVANTAGES

From Table 4.1 it can be seen that the Japanese have had a vast input into PSA technology during the past 20 years. This is as a result of an economic drive in Japan to reduce plant operating costs and to increase national productivity. The Japanese plants are exceedingly power sensitive due to the fact that all of the basic fossil fuels (oil and coal) that are used in Japan are imported which greatly increases

Company	No. of Plants	AVE Capacity	No. of Beds	Specific Power	Experience	Lead Time	Sieve Type	Range	Uses	Multinational	Prepared to deal in South Africa
		TPD		kWhr/ m_n^3	Years	Months		TPD			
Showa Engineering Company	85	19	3	0,4	15	8	Showa	0,15 - 90	Industrial	YES	YES
Nippon Steel	-	-	3	0,45	-	-	-	-	Industrial	YES	YES
Kofloc	-	-	2	0,52	-	1	-	0,03 - 0,25	Home / Medical	NO	YES
Sumitomo Seika	82	37	3	0,43	10	10	Sumitomo	1,2 - 137	Industrial	YES	YES
Taiyo Sanso	-	-	2	1,2	-	-	-	0,75 - 1,5	Home / Medical	NO	NO

TABLE 4.2 JAPANESE TECHNOLOGY BY MANUFACTURER (Bussiness information, 1991)

the cost of electricity. This power sensitivity may not suit the situation in South Africa where electricity is relatively cheap. Japanese process plant manufacturers are only willing to guarantee plant power and output performances if all of the mainline equipment is sourced from Japan. This will lead to problems associated with spare part availability and maintenance. It is also not altogether clear as to how much support a Japanese manufacturing company would be prepared to give to a South African company. Furthermore, the declining Rand value accompanied with the high import duties and surcharge tariff rates may make the importation costs of technology and equipment expensive.

From Table 4.2 and section 2.3.3.4, various PSA plant manufacturing companies have been discussed and reviewed in great detail. Showa Engineering Company (SEC), appear to be the most favourable and well known PSA plant manufacturing company, closely followed by Sumitomo Seika Chemicals. Both companies appear to have a very good product that could be imported and successfully installed in South Africa. Kofloc manufacture small two bed PSA oxygen systems that are very comparable to those from Airsep and BOC. They are however more expensive plant options.

4.2.4 BOC Group Technology

The available BOC group technology was reviewed as it developed during 1991 and 1992. The advantages and disadvantages are listed in table 4.3.

BOC has produced design packages for a small range of PSA plants. These are provisional designs only and it is the responsibility of the BOC group operating company that is producing the unit to examine the plant package and to complete the design. These designs are for both two bed pressure atmospheric plants and three bed vacuum plants.

4.2.4.1 Pressure Atmospheric Plants

Capital costs for the BOC group standard PSA plants were estimated and calculated from design packages and projected as installed plants in South

Africa for various plant capacities. The capital, power and maintenance costs are included to give a fair representation of the actual value.

BOC appears to use a significantly higher plant power than most other manufacturers. The calculated figures are highly theoretical figures only and are based on conservative designs. They will be verified shortly on the completion of a UK prototype that will produce 5 TPD. Expected plant specific power figures lie between 0,85 and 1,1 $kWhr/m_n^3$.

ITEM	ADVANTAGE	DISADVANTAGE
Research	On going , oxygen Centre of Excellence established. Receives input from OSK (Japan) GTC (USA) and all group operating companies.	May not catch up to world standards. Needs a large amount of group support to meet goals.
Technology	BOC have excellent engineers, scientists and technicians to establish a technology base.	BOC is very inexperienced.
Power	BOC plants may be more power efficient than American competitors (2 Bed)	BOC uses more power than the Japanese (3 Bed) PSA's
Trade	Easy trade with the United Kingdom.	
Mainline Equipment	BOC technology allows free choice of equipment, all which can be supplied locally.	Equipment selection may not be ideally suited to the process.
Manufacturers	Local manufacture will reduce costs.	Many manufacturers do not create a technology reference.
Support	Centre of Excellence promises excellent support.	
Cost		Weak Rand value, import duties and freight will increase costs.

TABLE 4.3

BOC TECHNOLOGY: ADVANTAGES AND DISADVANTAGES

BOC total plant operating costs, including capital costs, power costs and maintenance appear to be approximately 10% cheaper than those of Airsep. This can only be due to Alpha Air Separation having an additional profit margin on the capital costs of these imported units.

4.2.4.2 Pressure Vacuum Plants

Capital costs for the BOC group standard VSA plants were estimated and calculated from design packages and projected as installed plants in South Africa at various plant capacities. The capital, power and maintenance costs are included in order to give a fair representation of the actual plant value.

BOC appears to use a slightly higher plant power than most other process plant manufacturing companies. These calculated figures are theoretical figures only and are based on conservative designs. They will be verified shortly on the completion of the USA and RSA prototypes that should produce 30 and 10 TPD respectively. Expected power figures lie between 0,45 and 1.01 kWhr/m_n^3.

These total plant operating costs including maintenance, capital and power costs appear to match those of the other manufacturers as the product recovery on the BOC plants is higher than most others which will result in a lower plant capital cost.

4.2.5 Results

The results of the process survey for two bed pressure atmospheric plants, three bed pressure vacuum plants and the alternative forms of oxygen supply are discussed. The results of the plant manufacturers survey are also discussed. These two surveys have greatly increased the local engineering knowledge and viability of these non cryogenic air separation systems. Oxygen supplied from a high pressure gas cylinder has been excluded from this survey as the capacity is low and the costs are extremely high.

4.2.5.1 Plant layouts

The commercial evaluation discussed in sections 4.2.1 and 4.2.2 highlight that the two groups of non cryogenic oxygen plants have different economical factors. A bulk oxygen supply facility can also be supplied by three other methods, namely a liquid oxygen system, a low cost cryogenic oxygen generator, or a cryogenic air separation unit.

A bulk liquid oxygen storage facility will comprise of a TPV system. This TPV system is so called because it consists of a Tank (usually vacuum insulated and filled with pearlite to prevent product boil off. This is due to heat transfer between the vessel shell that is exposed to the atmosphere and the cryogenic liquid contained within the vessel. This heat transfer will heat up the cryogenic liquid and cause it to evaporate resulting in product loss.), a Pipeline and a liquid oxygen Vaporizer to vaporise the liquid into a gas. A TPV system is sometimes fitted with a cryogenic liquid oxygen pump if high a high instantaneous product flow rate is required. A TPV system is very dependant on the delivery of liquid oxygen from a nearby liquid oxygen plant, which is then also dependant on distribution and transport costs. These systems are very reliable and a vast majority of applications of oxygen are supplied by this method.

Cryogenic oxygen generators and Air Separation Units separate the atmospheric gases by using the different physical properties of the constituents such as their different vapour pressures and resulting different boiling points. The plant utilises very sophisticated machinery including high pressure gas turbines, high pressure oil free centrifugal air compressors, complex aluminium plate-fin heat exchangers and refrigeration units. Low purity (93% to 95% oxygen) cryogenerators are capable of producing gas at a supply pressure of up to 12 Bar(g). This eliminates the need for product oxygen compressors. This factor may sometimes increase the viability of the cryogenerator against that of a PSA or VSA system. Cryogenic plants are best utilised where large volumes of high purity oxygen is required. Cryogenic plants can also be utilised to provide small volumes of oxygen, below 100 TPD, however at these volumes the cryogenic oxygen plant becomes expensive.

To distinguish where the economical technology change over points occur for the supply of oxygen, it will be necessary to asses the exact requirements at the point of use. When creating a computer model to do this the same factors should be applied to all cases. Work done by the Author during the project execution has lead to the graph shown on Figure 4.3. This shows the expected Rand per ton price that would face the user for the four methods of oxygen supply.

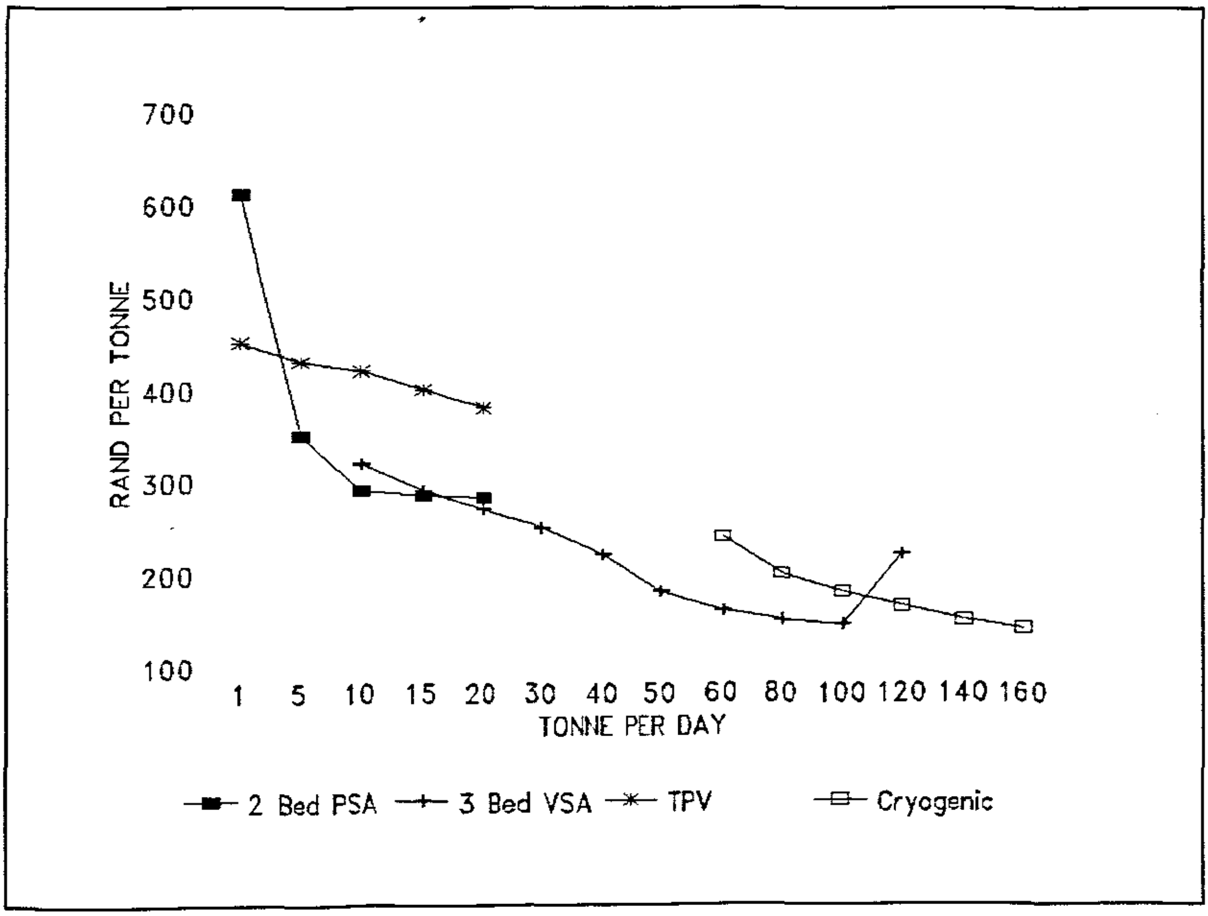


FIGURE 4.3

COST CURVE: CRYOGENIC, LIQUID AND PSA OXYGEN

From Figure 4.3 it can clearly be seen where the economical change over between two bed PSA oxygen, three bed VSA oxygen, liquid oxygen TPV systems and cryogenically produced oxygen takes place. This graph could be further projected to show that the traditional change over between a liquid oxygen TPV system and a cryogenic plant would take place at approximately 40 TPD. The PSA plant types fit into this gap and clearly provide a cost reduction with an associated change in mode of technology. The economical range for PSA oxygen plants starts at the swing over from liquid oxygen to two bed pressure atmospheric PSA plant layouts at approximately 3 TPD. The economical crossover between the three bed vacuum cycle plant layout and the two bed pressure atmospheric cycle plant layout lies between 15 and 20 TPD. Above 20 TPD, the three bed VSA plant layout would be used up to approximately 100 TPD, where at a supply greater than 100 TPD, an on site cryogenic oxygen system would be the most practical and economical installation.

Figure 4.3 contains guideline information only and is to be used as a starting point when an economical case study is to be performed. Each individual application must be independently examined and evaluated in terms of PSA system compatibility and the requirements for a back up liquid supply. A back up liquid installation could have a major cost implication to the installation costs and this would have a corresponding increase to the cost of the oxygen.

4.2.5.2 Manufacturers

Several companies in South Africa have started to market oxygen PSA technology. Little is yet known to be able to accurately assess the current state of the available technology. However, the known information is briefly outlined in Table 4.4. The most active companies are considered and briefly discussed.

Company	Technology	Type	Active in SA	World market share %	Experience (Years)	Comments
Fedgas	EVT Mahle (Germany)	3 bed VSA	Yes	2,9%	20	Fedgas have Nitrogen PSA systems running. It is assumed that they are investigating oxygen as well.
Air Products	Air Products (USA) Union Carbide	Two and Three bed	Unknown	11,5%	20	Air Products are not being active at all. They have world class technology and could easily become competitive.
Liquid Air	L' Air Liquide (France)	2 and 3 bed VSA	Unknown	3,0%	6	Liquid Air have just developed a Cryogenic Adsorption System to supplement their product range.
Alpha Air	Air Separation (USA)	2 bed PSA	Yes	1,9%	5	Alpha Air Separation appear to have a good product.
Ramfab	Oxxon (Austrian)	2 bed PSA	Yes	>0,1%	5	Ramfab units have not been successfully used at Foscor in Phalaborwa.
Afrox	BOC	2 bed PSA 3 bed VSA	Yes	4,5%	6	BOC group technology is rapidly developing.

TABLE 4.4 SOUTH AFRICAN PSA SUPPLIERS (EVT Mahle, Air Products, Liquid Air, Airsep, Ramfab and BO

Table 4.4 shows that Fedgas are considering PSA systems as they have successfully commissioned a nitrogen unit in a controlled atmosphere application in the Cape Province. Air Products have had international links with Sumitomo Seika Chemicals in the past and this would indicate that they have access to suitable technology. Very little information is known about Liquid Air, however in Europe their parent company L' Air liquide has begun marketing VSA technology. Ramfab is manufacturing small two bed PSA systems under licence to Oxxon, an Austrian company. These units capacity range is between 0,1 and 1 TPD oxygen at 95% purity (Oxxon, 1987). These units are largely used for scrap cutting with a limited amount of success, as the fuel gas (LPG or dissolved acetylene) requirements for welding and cutting increase when using oxygen with a purity less than 99,5%.

Alpha Air Separation is marketing the world's largest selling type of two bed PSA plant. They are the only gas company aggressively marketing oxygen PSA units and they are advertising monthly in the Engineering News (Engineering News, 1992). Their plants are manufactured in America and the installed capital costs, power costs and total running costs can compare favourably to the costs of traditional supply of liquid oxygen through a Tank, Pump and Vaporizer (TPV) system. Table 4.5 outlines a comparison between Alpha Air Separation and BOC group technology.

Table 4.5 shows that Airsep plants are technologically very advanced. Airsep is represented in South Africa by Analysis Management Systems (AMS) who have offices in two major centres in South Africa. Their representation does not appear to cover as wide a geographical area as the gas companies do, who are represented in all of the major centres. Monthly plant operating costs for Airsep and BOC plants have been calculated and reduced to rand per tonne values and these have been compared with each other. The result clearly showed that the BOC type plant would economically be more favourable for Afrox to market than the Airsep plants. BOC has a plant using a higher specific power than Airsep, while Airsep plants are marginally more expensive. This is probably due to the fact that BOC plants are supplied to Afrox at cost price, while Airsep units would contain a certain profit margin.

ITEM	AIRSEP	BOC
Research	Airsep designs based on Union Carbide (USA) designs. Their commitment to research and development is unknown.	BOC Oxygen PSA Centre of Excellence is committed to world class research. BOC has excellent laboratories and research personnel.
Technology	Airsep appear to be leading two bed plant design technology. Technology may date.	BOC will endeavour to meet and maintain all first world technology standards.
Power	Airsep PSA plants are relatively power efficient.	BOC uses slightly more power.
Mainline Equipment	Airsep predominantly American, which may cause problems associated with non metricated components.	BOC uses locally supplied items of equipment.
Support	Airsep is represented by AMS, with offices in Johannesburg and Cape Town.	Afrox has 36 branches nationwide, all capable of providing an installation and maintenance service to PSA plants.
Cost	The weak Rand / Dollar exchange rate will lead to increased prices.	Local manufacture could reduce costs and support local industry.

TABLE 4.5

COMPARISON BETWEEN AIRSEP AND BOC PLANTS

4.2.5.3 Oxygen Concentrators

During the course of 1992 several small companies, usually servicing the healthcare industry, introduced a range of oxygen concentrators for home, medical and hospital uses. These companies include Alpha Air Separation, Didier, Draeger, Air-Med and Art Medical Equipment.

Commonwealth Industrial Gases (a BOC subsidiary company in Australia) ignored similar occurrences in 1990 and as a result a large portion of its medical oxygen cylinder business was replaced by medical oxygen PSA units. A similar event could happen to the South African gas companies. Very little is known about oxygen concentrators in South Africa. Oxygen concentrators usually supply product at a few litres per minute at 80% to 90% contained oxygen (Puritan Bennett, 1990). They have been available for a few years but it is now expected that the market demand for these units will increase.

4.3 PLANT CONSTRUCTION AND PERFORMANCE TESTING

The pilot plant construction and performance testing forms a large part of the process understanding of the principals of adsorption technology. The information obtained from the operation and evaluation of the pilot plant are contained within this section of the text. Many recommendations are made that are related to improvements in the plant design and to modifications that should be incorporated into future VSA plant designs.

4.3.1 Pilot Plant Construction

All machinery and vessels on the pilot plant are skid mounted. This is to make the entire pilot plant transportable so that it can be moved from its commissioning and test site to a future customer site for providing an oxygen supply to an iron and steel mill. The pilot plant was then built on two skids, one for the three adsorber vessels and associated pipework and the second for the mechanical equipment. A Zozo hut (Portacamp) was used as a site office and control cabin. The oxygen product buffer tank and oxygen compressor were free standing items. The complete plant general arrangement drawing is shown in Appendix 3 and a photograph is shown in Figure 4.4.

This Figure shows the two main skids for the three adsorber vessels and the equipment and they can be seen completely installed in their relative positions. The low pressure oxygen product buffer vessel is on the left hand side at the back of the plant from where the oxygen gas is vented back into the atmosphere. The Roots blower acoustic canopy and the vacuum pump assembly can be seen on the front skid with the blower on the left hand side and the vacuum pump on the right hand side. In front is the zozo hut control room and all the instrument and electrical cabling can be seen on the cable racks between the zozo control room and the plant. The pipelines on the left are the cooling water supply and return pipes as well as the instrument air supply line. The 750 kVA transformer is on the right hand side of the control room and the low voltage (380 volt) motor control centre is located inside a separate cubicle in the zozo control room.

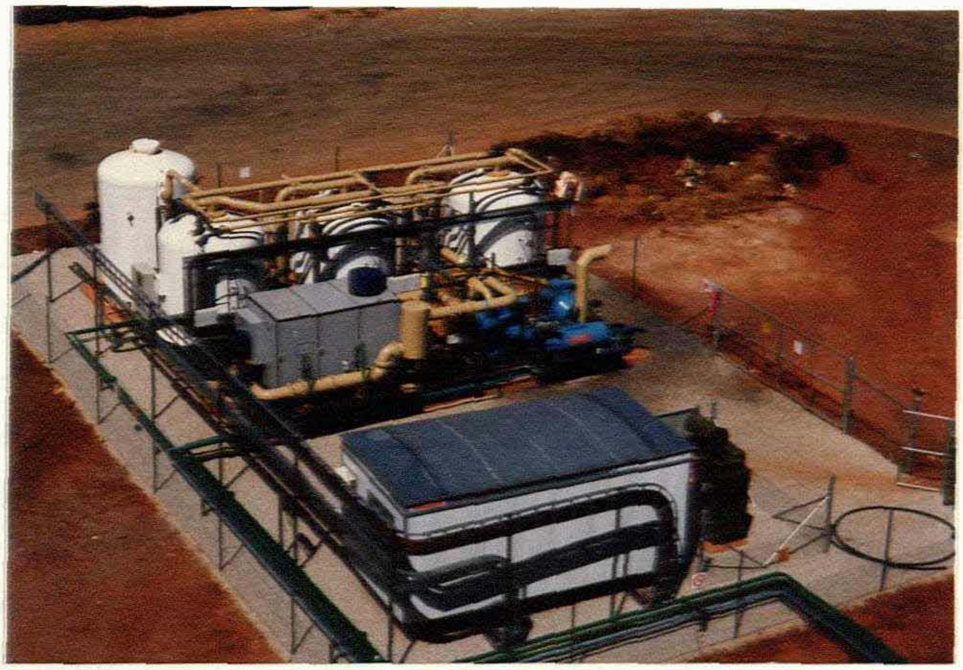


FIGURE 4.4
PILOT PLANT LAYOUT

<u>Vessel Skid</u>

The vessel skid had to be designed in accordance with the local road ordinance. This limited the height, width and length of the entire skid. In order to comply with the road ordinance specification the skid was design to incorporated the bottom pipework within the skid structure which resulted in keeping the height of the top pipework to a minimum. The structural design of the skid base was sub contracted to Jones and Wagner, consulting Civil and Structural engineers.

The vessels and pipework as discussed in section 3.3.2.3 were mounted onto the skid by IR&D. The entire unit was constructed at IR&D under "Grey Room" (Semi clean) conditions. The Fisher valves were all fitted with viton gaskets as the pipework was manufactured. The entire unit was pressurised to 2000 mbar (abs.) and maintained at this pressure for twelve hours and then evacuated to 200 mbar (abs.) and maintained at this vacuum condition for a further twelve hours to complete the pressure and vacuum leak testing. The choice of schedule 40 seamless pipe led to many complications during the plant manufacture that were mostly due to the mass of the process pipe fittings. This often resulted in the use of an overhead gantry crane for the movement and the positioning of several large elbows and Tee pieces. The thickness of the pipe wall required many capping runs to be laid onto the argon root run during the pipe welding procedure.

After the pressure and vacuum testing, the top pipework was disassembled and removed to allow access for the vessels to be filled. Alumina and molecular sieves are hazardous and all safety precautions had to be adhered to. These safety procedures included dust masks and eye protection. The molecular sieve was loaded using a 'sieve sack' which is a 1 m^3 polypropylene bag. Each bag held 4 drums of molecular sieve and when hoisted had a total mass of 500 kilograms. Each drum was individually weighed to determine the mass of zeolite molecular sieve installed into the vessels. Attached to the trunk of the sieve sack was a constant density filling chute which allowed an even distribution of the molecular sieve. The vessel filling arrangement is shown in Figure 4.5. An industrial vacuum cleaner was installed in the vessel during the filling to reduce the atmospheric dust levels.

The layers of alumina were added first to all the vessels and this was then levelled before the filling of the zeolite molecular sieves. After the zeolite molecular sieves had been loaded directly on top of the alumina, difficulty was experienced with the installation of the stainless steel screens, used to separate the zeolite molecular sieves from the top bed layer. Scotch pad discs supplied by 3M were successfully tested for oxygen compatibility and were then installed in the place of the stainless steel screens. These prevent top bed movement by separating the zeolite molecular sieves from the top bed layer. A small amount of top bed movement was detected on bed C, indicating that there was some degree of channel flow occurring where the scotch pad touched the vessel shell. A further alumina layer was then added above the zeolite molecular sieves as the ceramic balls, that were planned to be installed, contained a degree of moisture that may be detrimental to the zeolite molecular sieves (Norton, 1992). A vibrator was attached to the vessel after filling which allowed the beds to settle giving a greater packing density and the required volume of sieve was added. The top pipework was then replaced and the unit was pressurised to 1500 mbar (abs.)

for transport to the plant testing site in Pretoria.

FIGURE 4.5

VESSEL FILLING OPERATION

The packing density varied slightly to that required in the BOC design package. The effects of this difference were discussed in more detail in section 3.3.1. Snow shooting procedures and cyclone filling equipment should be investigated for future plant vessel filling operations. The molecular sieve crush strength is an important sieve characteristic that determines the viability of these filling processes, as a soft molecular sieve may be crushed to a fine powder during a snow shooting exercise. Snow shooting and cyclone filling usually results in a far greater packing density being obtained.

The vessel filling arrangement can be seen in Figure 4.5. The vessel filling took place in the workshops of Industrial Research and Development, utilising their overhead gantry crane. The sievesack and trunk are connected to the constant density filling chute which is about to be inserted through the vessel manhole and the molecular sieve would then slowly be discharged into the vessel.

Equipment Skid

The equipment skid was structurally designed by Jones and Wagner. This skid, shown in Figure 4.6, contained the Roots blower and the blower process pipework, the vacuum pump, the water separator, the vacuum pump process pipework and the instrument air buffer tank. Cooling water and instrument air were to be provided by pipeline from an existing adjacent Air Separation Unit plant cooling tower and feed air compressor at the plant test site in Pretoria.

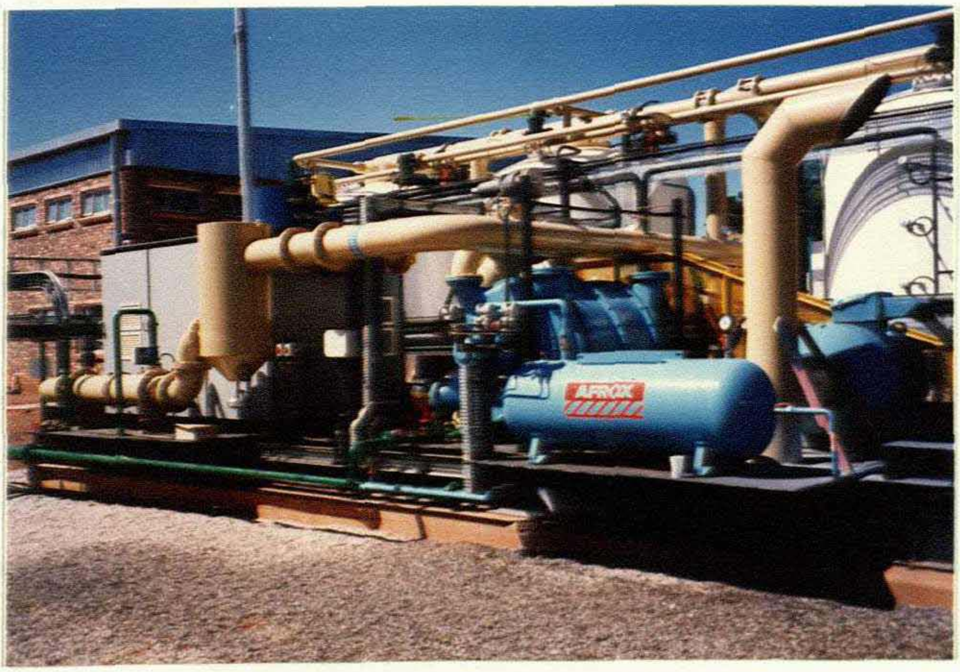


FIGURE 4.6
EQUIPMENT SKID

The Aerzen Roots blower was supplied with an acoustic canopy to reduce the noise pressure levels to comply to the legal legislation for noise protection devices (MOSAct, 1987). The entire construction of the Roots blower, the acoustic canopy and the associated pipework was contracted to Airgas Compressors, who assembled the unit on site and maintained responsibility of the machine until it had been commissioned.

The Torrvac liquid ring vacuum pump required a water and gas separator to be fitted. This was installed underneath the skid and allowed a water outlet to be fitted. The saturated waste nitrogen gas was vented away. Torrvac maintained the responsibility of the machine until it had been commissioned.

In Figure 4.6, the equipment skid can be seen standing on its parapet type concrete foundations. In the foreground is the instrument air buffer vessel and waste nitrogen vent. The vacuum pump and motor guards are situated behind the buffer vessel. On the right hand side the Roots blower acoustic canopy is shown. On the discharge pipework the aftercooler and condensate separator are shown. The instrumentation and electrical cable racks are also attached to the skid and will be transported along with the entire unit.

Instrumentation

The instrumentation panels containing computer equipment, chart recorders, alarm annunciators and other electrical equipment were housed in the Zozo control room. This allowed for the cooling of the instrumentation panels by using the room air conditioner. All cabling was routed through junction boxes to simplify the disassembly and assembly before and after transport.

The entire instrument installation was installed by Set Point Analytics and the control panels were supplied and manufactured by AC Controls.

Electrical

The entire pilot plant was to be fed from an existing 11 kV substation. This necessitated an oil flooded T-type fused switch to be installed on the 33 kV incomer to the sub station and a K-type fused switch to be added between the 11 kV sub station and the 750 kVA transformer (11 kV to 380 V) that was installed to supply the 380 volts required to run the process machinery on the VSA pilot plant. Fault load monitoring and protection devices were all added to enhance the integrity and safety of the electrical equipment. The 380 volt distribution board, including motor starters and circuit breakers, were all housed in a panel (supplied by AC Controls) and installed in a separate room in the Zozo hut. The entire electrical installation was completed by TJ Electrical Contractors.

Civil

The plant foundations took the form of concrete parapets to hold the skids. These foundations were kept to a minimum and were locally designed by Jones and Wagner. Civil construction was undertaken by a local contractor, Associated Transport and Excavators (AT&E). After the skids had been placed on the plinths the entire plant site was covered with 12 mm stone chips to a depth of 75 mm.

4.3.2 Experimental Procedure

The pilot plant was built to verify the BOC provisional design package and to determine the various operating parameters for this type of plant. It also served the purpose of gaining and establishing local engineering knowledge of atmospheric gas separation by adsorption processes. In order to obtain the required measurements a large amount of instrumentation had to be added. The desired experimental procedures and results are listed below.

4.3.2.1 Process

In order to asses the BOC process design the following factors are to be calculated and measured.

Product Recovery

The product recovery is defined as the mass of oxygen product as a percentage of the mass of oxygen entering the plant. In order to calculate this, the mass of the feed stream is calculated and from known quantities of oxygen in air the mass of oxygen in the feed is determined. The product mass flow rate is calculated and from the purity measurement, the oxygen mass flow rate is calculated. The initial mass flow rate calculations are calculated by a pressure and temperature compensated Chameleon flow computer and the product recovery is calculated on a lotus type spreadsheet using an IBM personnel computer.

Specific Product

The specific product is a molecular sieve characteristic that is verified.

Specific product is calculated as the volume of oxygen produced from the plant in normal cubic meters per hour per cubic meter of molecular sieve installed in the plant. The volume of sieve is known from the quantity of sieve installed into the vessels during the filling operation. The volume of oxygen produced is calculated from the mass flow rate calculations.

Pressure Profile

During commissioning and testing the actual plant operating pressure was plotted against the ideal pressure profile an ongoing basis by using a three pen chart recorder. The shape of the pressure profile gives a good representation of the actual operation being undertaken and will record the operating pressure during the part cycle process steps. This determines the ideal pressures for this particular plant and any deviations between the three bed profiles must be justified.

Valve Openings

As shown on the piping and instrument diagram (P&ID) in Appendix 2, there are manual flow control valves (valves marked CV3 to CV7) situated behind each automatic actuated valve. These valves control the amount of purge and backfill flow that is allowed into each adsorber vessel. These valve settings are determined and their affects on the pressure profile, product recovery and product purity are assessed through various valve setting trials.

Product Purity

The product purity is assessed for content of all gases. This is analyzed in a laboratory for oxygen, nitrogen and argon which forms the bulk of the product stream. The product stream is also analyzed for trace quantities of moisture and hydrocarbons.

Plant Temperature

The heat of reaction in the adsorption bed is measured on the three RTD's installed at various positions in the adsorber vessels. The temperatures affect the sieve characteristics and causes deviations from the expected plant performance due to a difference in atmospheric conditions.

4.3.2.2 Mechanical

The integrity and suitability of the mechanical machinery, selected to provide the process parameters, are assessed. In order to evaluate these the following measurements were noted.

Pressure Equipment
The selected Aerzen blower must perform under conditions of cyclical loading. This unit produces a large noise pressure level which is suppressed with an acoustic canopy. Noise levels are measured to determine compliance with MOSact legislation and should be less than 85 dB(A), at a distance of 1 meter. Power requirements in terms of maximum demand and kilowatt-hour units are measured to assess the calculated specific power against actual. This determines the factors for scaling of plant specific power figures.

Vacuum Equipment
The Torrvac liquid ring pump must perform under conditions of cyclical loading. The implications of the liquid ring collapsing under the changing pressure conditions are assessed and the capacity of the separator is verified. The vacuum pump noise level is measured in order to comply with legislation. Power requirements in terms of maximum demand and kilowatt-hour units are measured to compare the calculated specific power against the actual. This determines factors for the scaling of plant specific power figures.

Vessels and Pipework
There were few measurements taken on the vessels and pipework. The bed pressure profiles will be plotted against each other to give an indication of the cyclical changes in the three adsorber vessels. All flanges and welds were checked for leaks and cracks during commissioning. Gaskets were all checked for leakage and system compatibility.

4.3.2.3 Control and Instrumentation

The pilot plant control and instrument system is evaluated for its adequacy to provide the measurements required in sections 4.3.2.1 and 4.3.2.2. Instrumentation is assessed on the choice of valve types and main instrument selection discussed in section 3.3.3.2. The variable speeds on the chart recorders are set and the PLC program is verified and modified to suit process control and operating parameters.

4.3.3 Results

The design, expected and actual results of the Pilot Plant performance are shown in Table 4.6. The design figures are the values given in the BOC design package, whilst the expected are the performance figures calculated in sections 3.3.2.1 and 3.3.2.2. The actual figures are measurements taken from the pilot plant during the plant performance testing. This shows several deviations which are individually discussed with recommendations in the following sections.

Item	Unit	Design	Expected	Actual
Specific Product	$m_n^3/hr.m^3$	13,4	13,4	14,6
Product Recovery	%	51,8	51,8	55,5
Product Purity	%	93	93	93
Product Flow	m_n^3/hr	313	313	341
Product Temperature	°C	25	25	25
Product Pressure	mbar(abs.)	1250	1250	1231
Specific Power	$kWhr/m_n^3$	0,45	1,01	0.93
Noise	dB(A)	103	85	61-89

TABLE 4.6

PILOT PLANT PERFORMANCE RESULTS

4.3.3.1 Process

The following results were recorded on which an evaluation can be made to determine the integrity of the plant process design.

<u>Product Recovery</u>

The product recovery during the plant commissioning varied widely from 39,4% to 55,8%, depending on the plant operating conditions. It was found that the product recovery increased with an increase in the purge valve opening and with an increase in the plant pressures.

The product recovery in the actual operating case appears to exceed design conditions by 6,7%. This can be as a result of several factors including a degree of over design on the plant, or because the top bed voidage is very low. A low top bed void area helps to recover most of the useful product. It was not expected to achieve such a high product recovery as the mass of VSA II zeolite molecular sieve in the vessel was below the design level. As a result of this, the top layer of Alumina occupies more space in the top dished end and this reduces the top voidage area. The most likely factor is probably due to an over design on the amount of molecular sieve to be installed.

Specific Product

The specific product varied during the plant commissioning between 9.4 $m_n^3/m^3.hr$ and 15 $m_n^3/m^3.hr$ depending on the plant operating conditions. It was found that the specific product decreased as the product purity decreased. Low specific product measurements were accompanied with low product flow and high specific power. The specific product improved as the product recovery improved.

The specific product in the actual operating case appears to exceed the design case by approximately 9%. Similarly to the product recovery discussed above, an over design condition on the VSA II zeolite molecular sieve loading giving an increased product flow rate can be an explanation for this phenomenon. The specific product was expected to be low due to the low mass of molecular sieve loaded to the vessels and was expected to be inversely proportional to the product recovery. These results clearly show that this is not the case in practice.

Pressure Profile

The pressure profile varied from the ideal pressure profile during commissioning as the relative changes in the plant operating mode were undertaken.

The most significant deviation is that the pressure decreased during the backfill step, rather than remaining constant. This is due to a high backfill rate and this can be controlled by either the product take off valve, CV7, or the backfill valve, CV3. The pressure profile at plant operating conditions plotted onto the chart recorders is shown in Figure 4.7. The deviations of these figures can clearly be seen when these profiles are compared to the ideal pressure profile as shown in Figure 3.9. The current operational chart speed is 25 mm meter per minute on the Y axis and the pressure scale on the X axis is for a full range from 0 to 1500 mbar (abs.).

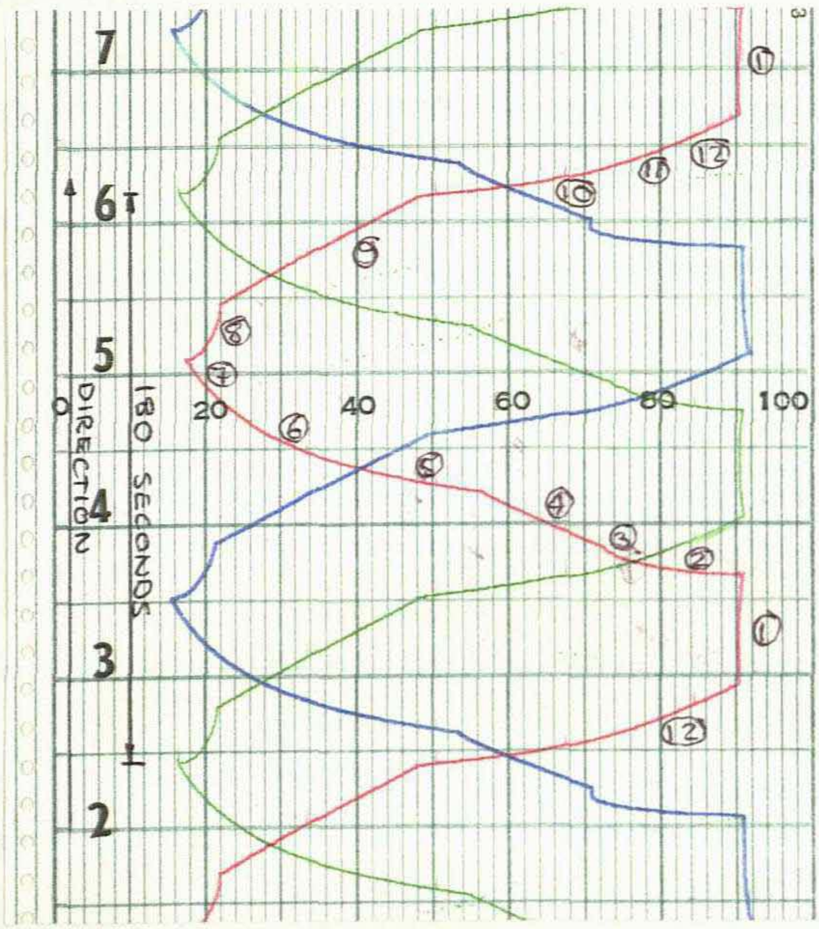


FIGURE 4.7
BED PRESSURE PROFILES

The plant outlet pressure in the operating mode is approximately 1,5 % lower than that in the design case. This can only be explained by the fact that the plant pressures being slightly low throughout the entire cycle. From experience gained by using these instruments, there is usually a 3% measurement tolerance applied to absolute pressure transmitters and the pressure deviation in well within this range.

In Figure 4.7, a print out from the Chino chart recorder is shown, where the continuous difference in the bed pressure profiles for each of the three adsorber vessels are indicated. The deviation in the minimum and maximum pressure level of the three adsorber vessels is due to a faulty pressure transmitter as discussed in section 4.3.3.3. The pressures in beds A, B and C are represented with the red, green and blue profiles respectively. By following the profile of bed A and the cycle sequence chart shown in Figure 3.15, the pressure profile is explained. The part cycles from step 1 to step 12 are indicated on the pressure profile. There is a small offset on the location of the three pens on the chart recorder, so the three graphs should not be read simultaneously. This offset is approximately one millimetre and in order to evaluate the simultaneous bed pressures the respective pressure profiles should be read one millimetre apart, along the Y axis.

Step 1, 34 seconds	Bed A is currently on production and is providing a portion of the product quality gas to the product buffer vessel and the remainder is being used to backfill adsorber vessel B.
Step 2, 6 seconds	Bed A begins the depressurisation step. Bed A still provides product to the product buffer vessel whilst the top of bed A is equalised with the bottom of bed B.
Step 3, 3 seconds	Bed A has the same status as in step 2, however the feed air is diverted from bed A to bed B.
Step 4, 17 Seconds	Bed A continues to depressurise by providing a flow of purge gas to vessel C.
Step 5, 34 Seconds	Bed A is now exposed to the vacuum pump and the bed pressure is reduced.
Step 6, 6 seconds	Bed A has the same status as step 5.
Step 7, 3 seconds	Bed A is evacuated to its final vacuum level.

Step 8, 17 seconds	Bed A begins the repressurisation process by receiving a flow of purge gas from adsorber vessel B.
Step 9, 34 seconds	Bed A is no longer exposed to the vacuum pump and continues the repressurisation process by receiving a flow of backfill from adsorber vessel C.
Step 10, 6 seconds	The bottom of bed A is equalised with gas from the top of bed C.
Step 11, 3 seconds	Bed A still receives an equalisation flow from bed C and the air feed is diverted to bed A. Bed A pressures reach production pressures.
Step 12, 3 seconds	Bed A is pressurised to the maximum production pressure and provides product quality gas to the product buffer vessel. The cycle will then repeat itself continuously.

Valve openings

The throttle valve settings were varied during the plant commissioning in order to achieve the most efficient plant operating mode. It is always important to remember that in process engineering an adjustment to something will eventually affect everything. The throttle valves were therefore adjusted very slowly to achieve the desired conditions. The throttle valves have the largest impact on the bed pressure profile. As there were no actual design conditions listed for the throttle valve openings the openings are highly variable and may be operated manually to alter the plant operation mode.

In the ideal operating condition the flow control was achieved by setting the manual throttle valves as follows:

Backfill Valve (CV 3) : 30° Closed
Purge Valves (CV 4A/B/C) : 45° Closed
Equalisation Valves (CV 5A/B/C) : 45° Closed
Product Valve (CV 7) : 25° Closed

The manual throttle valve system worked well and the desired flow conditions were achieved. It would be useful in the future to operate similar plants with a system of butterfly valves that incorporate positioners to control the valve opening. This would require the plant PLC to monitor the bed pressure and to open the valve according to the bed pressure profile. This would allow the plant to closely follow the ideal bed pressure profile with any deviations being automatically corrected by the plant PLC. A positioner controlled butterfly valve would eliminate the need for the manual flow control valves, as the positioner would perform the flow control function. Valve positioners are however more expensive than manual throttle valves.

Product Purity

The plant product purity varied widely during commissioning between 92,3% and 93,9%, depending on the plant operating conditions. The oxygen product flow rate had the greatest effect on product purity. The plant product purity was plotted on a chart recorder and is shown in green in Figure 4.8. This chart printout shows that the product purity increases when the plant production is high and when the air to plant flow rate is low. This is at the maximum pressure condition in the adsorber vessels and is also at the operating position that is closest to the design conditions.

A sample of the product oxygen gas was drawn from the oxygen product buffer vessel using a gas syringe. This sample was then analyzed in the Afrox special gases laboratory in Germiston. The results obtained are shown in Table 4.7.

Item	Unit	Result
Oxygen	%	93,2
Argon	%	4,1
Nitrogen	%	2,7
Carbon Dioxide	vpm	<10
Carbon Monoxide	vpm	<10
Total Hydrocarbons	vpm	10

TABLE 4.7

PILOT PLANT PRODUCT PURITY

The pilot plant achieved a higher maximum product purity than the 30 TPD prototype in the USA, also built to verify a 30 TPD provisional BOC design package. At all times this product purity was maintained as close as possible to the plant design purity. This required a large degree of fine tuning to ensure that the balance between the product off take rate and the rate of backfill gave the plant optimum purity.

Plant temperatures

The plant temperatures did not vary widely during the plant commissioning. The plant bed temperatures can be controlled by adjusting the top to bottom equalisation flow rate through valves XV5 A/B/C. An increase in the valve opening resulted in the cold spots to warm up to above 0°C. A cooling effect was experienced in the oxygen product flow. This can be explained by the development of a cold spot in each vessel in the bottom region of the VSA II zeolite molecular sieves just above the alumina layer. The cold spot temperature was not very significant as it was measured between -1°C to 5°C. Cold spot temperatures have been recorded on other similar plants up to -50°C and at this temperature, combined with the cyclical pressure loadings, the vessel shell can rupture. The effects of ambient air temperature changes caused the plant purity and operating pressures to fluctuate. These were corrected by the addition of pressure control equipment and is discussed in greater detail in section 4.3.3.2.

The air to plant temperatures were at all times exceptionally low. This was due to the cooling tower performing well and providing cooling water at an approach temperature of 2°C above ambient conditions. It was necessary to adjust the water flow through the aftercooler to regulate this temperature and control was not always successfully achieved. An automatic temperature control valve should be installed on any future plants to monitor the gas temperature and then to regulate the water flow rate to the aftercooler in order to maintain a constant air feed temperature. The optimum air feed temperature is the temperature defined by the adsorption isotherm and in the case of the pilot plant, the optimum temperature is given on the isotherm shown in Figure 3.3 as 25°C.

Summary

The plant operated well and achieved the design operating conditions within a minimum period of time. There was a major cause of concern regarding the performance of the plant due to the incorrect packing density of the molecular sieves. It was initially expected that the plant may not actually achieve its design parameters, as the mass of molecular sieve was approximately 4% below the design. However, careful process optimization resulted in these difficulties to be overcome and enabled the plant to achieve the design requirements.

The element of plant production capacity needs to be addressed in terms of process over design capacity. The plant produces approximately 7% above design and when the volume of sieve is considered it appears that the process design is exaggerated by approximately 10 %. This over design can have a huge impact on the plant capital costs. By applying the 0,6 rule, as discussed in section 4.2, to the capital cost breakdown shown in Appendix 1, it appears that the cost implications of this over design amount to R 184 752,00 and this is shows a 5% cost implication which is a significant amount of money.

The plant achieved a stable operating condition and maintained that condition during the performance testing. The plant achieved an acceptable purity and the verification of the total hydrocarbon count may make a VSA plant a more favourable oxygen supply option to an application such as ozone production where low hydrocarbon levels are critical. The plant required little operator input and could quite simply operate as an unmanned plant. The simplicity of the process indicated that plant mode adjustments and output scale up modifications would be relatively simple. This plant proved that the successful implementation of commercial oxygen VSA process plants will be possible in South Africa in the near future.

4.3.3.2 Mechanical

The following results were recorded on which an evaluation can be made regarding the operational integrity of the mechanical items.

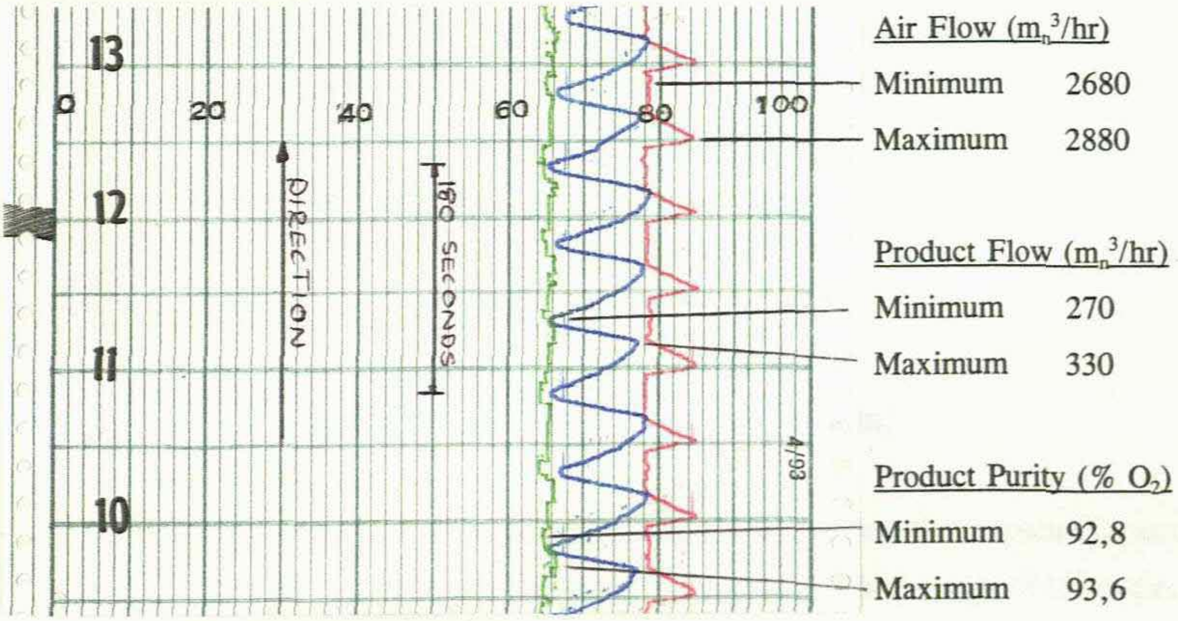
Pressure Equipment

The Aerzen Roots blower was commissioned with minimum effort. Several commissioning problems included the response speed of the recycle equipment, the calibration of the safety valve and general instrumentation problems. Following from all of these, the air blower was run for 24 hours prior to the process commissioning.

The air blower appeared to perform well during the initial stages of commissioning and performance testing. The blower delivered the required volume of air to the pilot plant. The ambient temperatures did not exceed design conditions during the performance tests so it is still to be ascertained as to whether the blower delivers 2690 m_n^3/hr at the design conditions. The blower aftercooler equipment performed well under the operating conditions and delivered air to the pilot plant at a maximum temperature of 25°C. The moisture condensate separator performed its functions well by continuously dripping the condensate out of the automatic drain valve which prevented water carry over onto the molecular sieve beds.

The Roots blower is a positive displacement machine and will deliver the inlet air to the plant. It was noticed that at night, when the atmospheric temperatures were cold, or lower than 5°C, that a larger air flow rate was experienced. The air to plant flow is shown in Figure 4.8. The air blower design inlet temperature condition is 25°C, and lowering this by 20°C results in a 7% increase in the air flow rate due to the increase of air density associated with a decrease in the air temperature. This increased air flow rate caused the plant pressures to increase and a reduction in both the product purity and product recovery was experienced. This problem was solved by installing an automatic pressure control valve on to the blower recycle equipment which would recycle a portion of the flow when the plant pressures were high. Through a process of trial and error, the pressure controller reached its optimum set point at 1350 mbar (abs.).

The plant purity, the air to plant flow rate and oxygen product flow rate are shown on Figure 4.8 in green, red and blue ink respectively. The X axis is using three separate scales as shown on the chart printout and the current chart speed is 10 mm per minute. The air to plant flow rate increases when the blower outlet pressure is low, which is at the start of the pressurisation steps. The flow decreases as the pressure in the blower increases due to the blow back of air through the clearance gap between the rotors and the machine casing. A high product oxygen flow rate is also experienced during the high airflow condition due to low flow velocities entering the product buffer vessel discharge pipework causing lower plant pressure drops. This product flow condition is variable because there is no back pressure on the VSA plant as the buffer discharge and oxygen product is vented back to the atmosphere. In practice it is expected that the product flow will be more constant due to some back pressure being caused on the system by the oxygen application.



Air Flow (m_n^3/hr)
Minimum 2680
Maximum 2880

Product Flow (m_n^3/hr)
Minimum 270
Maximum 330

Product Purity (% O_2)
Minimum 92,8
Maximum 93,6

FIGURE 4.8

PLANT PURITY AND FLOW RATES

During the second week of plant performance testing the air blower resonator developed a small crack along a weld seam which, led to a reduction in the air flow. The crack was caused as a result of apparent bad manufacturing practice. The welded seam had been ground away with an angle grinder to give the resonator an aesthetic appearance, but in the process of doing so, the weld penetration was also removed leaving a weak point where the stresses accumulated resulting in weld failure. The weld was repaired by Airgas Compressors and after three similar failures the entire unit was stripped out and the weld seams were butted together with 50 mm angle iron to give the structure more strength.

The blower performance under cyclical operating conditions did not present any major problems. A small problem created by the slow recycle equipment was rectified by adding a spring to the valve actuator and adding volume boosters and larger solenoid valves to the instrument air impulse lines. The blower recycle valve is shown in Figure 3.8 as XV91. The blower recycle valve was sized at 100 mm and this allowed a full flow recycle at 1240 mbar (abs.). A larger recycle valve would allow the plant to recycle at a lower pressure, whereby less power would be required during plant idle periods. This had a severe impact on the plant power at turndown, as the power absorbed during recycle is larger than the average power over a complete cycle. The absorbed power recorded on the powerview meter gave the following readings:

At start of cycle : 37,54 kW
At end of cycle : 68,38 kW
Specific Power : 0,198 kWhr/m_n^3

The total power demand over the cycle is higher than that expected in section 3.3.2.1 and these differences can be explained. At the start of the cycle, air is admitted to the bed at 1100 mbar (abs.), which is 100 mbars higher than in the design case and the pressure increases rapidly to a maximum pressure of 1350 mbar (abs.). This high pressure point reached in practice is 150 mbars lower than the expected pressure. These pressure differences are necessary to operate the plant at the optimum output levels. This results in the power demand at the start of the cycle to be proportionally higher than expected and the power absorbed at the end of the cycle is lower than what had been initially expected. The plant specific power is also higher than expected as the nett pressure increase is not linear. This can be seen on the chart recorder printout shown on Figure 4.7. The product output flow is also marginally higher than design, as discussed in section 4.3.3.1 and this will have the effect of improving the plant specific power. The power factor on the motor driving the air blower ranged between 0,65 and 0,76. The power factor is low when the absorbed power is low which corresponds to low plant pressures. This low power factor will lead to a high maximum demand to be incurred for this type of plant.

The acoustic canopy successfully decreased the noise pressure level to well below 85 dB(A). The noise pressure level was recorded by the South African Bureau of Standards as 79,4 dB(A) at the acoustic canopy (SABS, 1993). There were no problems present during the performance testing indicating high vibration levels as the machine vibrations were absorbed by the rubber anti-vibration mountings that were installed during the plant construction.

Vacuum Equipment

The Torrvac liquid ring vacuum pump was commissioned with minimum effort. Several commissioning problems included vacuum breaker calibration, belt tension, water throughput and low water flow rate alarm indication. Following from this the vacuum pump was run for 24 hours before the process plant commissioning.

The vacuum pump appeared to perform well during the initial stages of plant commissioning and performance testing. It is very difficult to measure the flow rate under vacuum conditions so the method of ascertaining the pump performance was to monitor the pump in an actual operating condition. The vacuum pump successfully managed to evacuate and regenerate the molecular sieve beds to the required absolute pressure levels as indicated by the VSA II molecular sieve isotherms shown in Figure 3.3 and the actual pressure level can be seen on the chart recorder printout shown in Figure 4.7. The water flow rate to the vacuum pump was determined by adjusting the water inlet gate valve until the pump vacuum level was correctly set. An adjustment of the water flow rate through the machine will have an affect on the fluid temperature rise, the final vacuum level and the power requirements. An increase in the water flow rate will result in a greater vacuum level, increased power demand and less temperature rise across the machine. The water flow rate could not be measured as a flow rate indicator had not been installed. The water flow was manually adjusted until a temperature rise of 6°C was experienced across the machine, giving a suitable vacuum level and power demand.

The vacuum pump performed well under cyclical operation from a process point of view. Under these conditions it managed to fully desorb and effectively regenerate the molecular sieve beds. The liquid ring constantly collapsed on change over between the beds which exposed the water to an instantaneous absolute pressure change of 610 mbars. This discharge of water lead to flooding of the cooling water sump and water carry over down the waste nitrogen vent. Torrvac had initially recommended that an 800 litre water sump be fitted, but it was more economical to install a 1200 litre sump to the equipment skid base and to utilise existing steel sections into the sump design. A gravity return line was installed to return this water to the cooling tower but an insufficient pressure head did not allow this water return system to operate. No efforts were made to prevent the liquid ring from collapsing due to the fact that the pump is not designed to handle instantaneous inlet pressure changes and the pump was performing its process duties well. Later, an air operated Wilden vane type water pump was added to this sump, to keep the sump empty and to prevent water carry over down the waste nitrogen vent. This water was pumped away and discharged into the plant cooling tower sump.

The vacuum pump idle power was high and as in the case of the air blower, the idle air inlet valve was incorrectly sized at 150 mm, which greatly restricted the air flow rate through a valve this size. The idle air inlet is shown as valve XV71 in Figure 3.8. The idle air inlet valve will allow atmospheric air to enter the vacuum pump during periods when the pump is running but is not actually evacuating the molecular sieve beds. A larger valve would result in less power to be absorbed during these periods, especially during plant turndown. The absorbed power recorded on the powerview meters gave the following electrical readings:

At start of cycle : 207 kW
At end of cycle : 248 kW
Specific power : 0,751 $kWhr/m_n^3$

The vacuum pump power varies quite significantly from the expected as calculated in section 3.3.2.2. The main difference is due to the fact that the process parameters are slightly different to that on which the calculation is based. From Figure 4.7, it can clearly be seen that the pumpdown curve is not linear and therefore our assumption on which the calculation is based is incorrect. However, as the pumpdown is achieved in a short time, the result indicates the average vacuum pump power integrated over a complete cycle is close to the expected power at 238 kW. The difference between the assumption on the shape of the curve will have resulted in the machine being slightly oversized for the process, however this is not detrimental to the process as the pump will then achieve a greater vacuum and have the result of a better molecular sieve regeneration. The vacuum pump specific power is reduced due to the high product flow rate, explained in section 4.3.3.1. The vacuum pump power factor varied between 0,82 and 0,89. This power factor is not significantly low but it will still incur a high maximum demand charge.

The vacuum pump noise pressure levels were recorded by the South African Bureau of Standards. The noise levels varied around the pump from 82 dB(A) at the pump to 89 dB(A) at the V-belt drive. These noise levels were higher than the expected noise levels. These noise levels could be caused by the V-belt vibration and this might be reduced by replacing the existing V-belts with another matched set.

The vacuum pump experienced problems associated with vibration as a result of the V-belt vibrations. The entire machine with a mass of nearly 1500 kg caused the equipment skid to vibrate. The skid was eventually braced with steel wedges. Stays were added to most upright and vertical pipes to prevent them from shaking and to prevent fatigue failures form occurring. Similar units should in future be mounted on to solid concrete plinths and be bolted down.

Vessels and Pipework

The three vertical adsorber vessels built by IR&D performed well during the commissioning and performance testing stages of the pilot plant. The vessel sight glasses allowed for the early detection of top bed fluidisation. A serious problem would have arisen if this had not been detected on the initial plant start up. The torrispherical dished ends were a good selection as the reduction in the top bed voidage area helped to contribute to the good plant performance as indicated in section 4.3.3.1. The vessel skid was subjected to a degree of vibration as a result of to the interconnecting pipework to the machinery skid. This was reduced with the installation of steel wedges.

The adsorber vessels were built to achieve the volume that was required to contain the zeolite molecular sieves as outlined by the sieve selection. It may be necessary to increase the vessel diameter to allow for the correct mass of molecular sieve to be installed as the filling density was lower than expected. This will not be necessary if alternative filling methods produce satisfactory filling densities.

The process pipework underwent final leak testing and pressure testing on site before the plant was started up. No significant leaks were detected. It was necessary to modify some of the discharge pipework on the oxygen product outlet in order to install CV 81 as a manual flow control valve to control the final oxygen output flow rate through the butterfly valve XV81 shown on Figure 3.8. The choice of viton gaskets proved to be a good as the flange to flange sealing was excellent. The pipework not exposed to vacuum conditions was fitted with Compressed Asbestos Fibre (CAF) gasket material which is approximately 90% cheaper than viton.

Summary

The selected mechanical items all performed well under the actual operating conditions. The selection of machinery type gave few problems and it is most likely that similar plants will utilise Roots blowers and liquid ring vacuum pumps rather than the alternative machinery types as discussed in section 3.3.2. The most major hazard was caused by the vacuum pump water discharge. The problem was rectified with the addition of a pneumatic sump pump which allowed the water to be pumped away to the cooling tower sump. The addition of a product oxygen compressor can quite easily be accomplished as the necessary pipework is in position for this system to be installed.

The overall plant power figures recorded at the plant distribution board on the motor control centre gave the following readings:

Total Units	:	311 kWhr
Maximum Demand	:	324 kVA
Power Factor	:	0,96 Lagging

These power readings vary slightly from the power readings on the powerview meters. The readings taken on the plant distribution board meters include copper and iron losses in the transmission cables, power for lights and air conditioner in the site office, power for the control panels and for the plant computer and recording equipment. The addition of all the extra devices for power usage has the nett effect of increasing the plant power factor which reduces the apparent electricity costs for the plant operation. From these readings the overall plant specific power is calculated to be approximately 1,04 $kWhr/m_n^3$, depending on the plant operational mode.

From the adsorber vessel pressure profile it will be possible to accurately calculate the theoretical power demand for a Roots type vacuum pump. This area of total plant power needs to be addressed as the power consumption of the three bed pilot plant is far higher than the theoretical power consumption of a plant operating on the two bed pressure atmospheric cycle. This means that the three bed VSA plant systems may not be economically viable when operating with liquid ring vacuum systems. The theoretical vacuum pump calculation was estimated by the BOC Group Technical Centre (GTC) as 0,285 $kWhr/m_n^3$. By substituting this value to the total plant power figures, a plant specific power of 0,57 $kWhr/m_n^3$ is obtained, which is favourable when compared with the expected two bed pressure atmospheric plant power.

The total plant noise pressure level varied between 61 dB(A) and 89 dB(A). The lowest noise zone is in the plant control room, where the noise levels were recorded as 61 dB(A). The noise levels were higher than expected as the vacuum pump V-belts were vibrating highly and causing excessive noise levels. The high noise levels resulted in certain areas of the pilot plant to be zoned as noise zones, where hearing protection should be worn. This is not detrimental to this plant in any way, as it has been designed to operate as a fully unmanned plant.

The plant operating parameters were well defined by the choice of mainline equipment and performed well for all practical commissioning and performance testing purposes.

4.3.3.3 Control and Instrumentation

The plant control and instrumentation were evaluated separately to give a complete understanding of the plant performance parameters.

Plant control

The plant control was achieved with the use of a Texas Instruments model 435 PLC. This PLC continuously recorded the plant bed pressures and allowed the plant to function to its normal operating cycle. There were many problems experienced due to the PLC program being based on timers rather than events. This meant that if one timer was changed the entire plant operating cycle went out of sequence. This had severe implications since it is always possible to expose the air blower discharge to the full vacuum inlet on the vacuum pump. This did occur on one occasion during the plant commissioning and the result was that the plant conducted an emergency shutdown only after the air inlet filter had been damaged and the vacuum breaker operated efficiently. Further shortcomings of the plant PLC was that the PLC did not have a built in freeze function. This freeze function would have allowed field checking of the actual valve position during times of plant failure. This freeze function is a feature that is available on the Texas Instruments model 535 PLC and this model PLC should be used in future.

The plant alarm and annunciator system worked well allowing a first up system to be implemented. This first up system would indicate the cause of a system trip. When the plant trips, several other alarm tiles will be activated during the plant shutdown, but when the plant trips are acknowledged and reset, the tile indicating the initial trip will remain illuminated, showing the trip cause and allowing the plant failure to be analyzed. The plant tended to trip out on a low instrument air pressure which was as a result of fluctuating instrument air supply from the existing ASU. The ASU main air compressor was repaired and the problem did not re-occur.

Plant flow calculations were accurately calculated on the chameleon flow computer. The Chameleon flow computer required some recalibration as the constants that were programmed into the unit were incorrect and the outputs were approximately 10% low. The daily production figures and calculations of product recovery, specific product and specific power were completed on a Lotus spreadsheet system with information obtained from the Chameleon flow computer.

The process operated switching valves were all fitted with proximity switches intended to give a positive feed back to the plant PLC. The PLC was intended to trip the PSA plant if a valve should not close within the required time. The proximity switches were not very reliable and the trip function was removed after several unnecessary plant trips where there were no apparent problems. The trip function was then changed to an alarm function. A valve failure would then be detected by a plant permissive trip as discussed in section 3.3.3.1 and shown on Table 3.4. The plant then operated successfully without any further valve problems.

Plant instrumentation

The plant instrumentation supplied and installed performed well in the actual operating conditions. The major problems were associated with the absolute pressure transmitters.

The absolute pressure transmitters were calibrated with a range of 0 to 1500 mbar (abs.). The calibration of these instruments allows a 0,1 % deviation on the span and zero set point. The pressure transmitters were not altogether accurate over this range as was shown on the bed pressure profiles plotted one against the other. The pressure transmitter failures were related to an oxygen compatible oil called Flurolube that constantly evaporated from the pressure transmitters. The pressure transmitters were replaced and then recalibrated two more times before the units were deemed to be accurate for process measurement. These pressure transmitters were used to give signals to the plant PLC and were used for the detection of the bed permissive trip errors. It was therefore vital that these units were accurate.

The Fisher actuated control valves performed exceedingly well under the loads placed upon them. The solenoid valves that operated the valve actuators were fitted with exhaust silencers to prevent excessive noise emissions on valve changeover. These subsequently were removed as they soon became clogged up with dust from the dusty atmosphere near the Iscor site in Pretoria. The operation of the process switching valves was displayed the plant mimic located on the plant control panel. This mimic received a signal from the proximity switches on each of the switching valves and showed the valve openings on a process mimic. This mimic proved to be exceptionally helpful during the plant commissioning process.

The Servomex 1100 paramagnetic oxygen analyzer performed its function well. The autocalibration function would check the analyzer range between a sample of calibrated analysis gas at 99,99% oxygen and atmospheric air. The pilot plant would trip whenever the analyzer performed this calibration function as the pilot plant PLC would receive an oxygen purity signal of 20,9% due to the oxygen purity in air. This autocalibration function was then removed and the analyzer was calibrated manually.

The local and remote pressure gauges, temperature gauges and flow metering devices all performed to a suitable standard for plant operational purposes. These items were all supplied with calibration certificates, however, their accuracy is not as critical as the switching valves and pressure transmitters.

Summary

The scope of instrumentation supplied to this pilot plant vastly exceeds the amount of instrumentation that would be required on a commercial unit. The scope of instrumentation gave all the required plant measurements. Once all of the instrumentation commissioning problems had been solved the plant instrumentation performed well.

For future plant construction, some of the instrumentation equipment installed onto the pilot plant will no longer be necessary. The flow computer will be replaced with a PLC function, linked into a personal computer with a screen display. This will replace the plant process mimic. The personal computer will also allow for the addition of a telemetry system to make provision for remote plant monitoring.

The reliability of the selected instrumentation contributed greatly to the success of the pilot plant commissioning and performance testing.

5 CONCLUSIONS AND RECOMMENDATIONS

5.1 PSA TECHNOLOGY

5.1.1 Theoretical Considerations

The theoretical considerations of PSA technology discussed in sections 3.1 and 3.2 have given a good understanding of the mode of technology utilised in separating oxygen from the atmosphere by pressure swing adsorption. The technology is well established in many Eastern and Pacific rim countries and because of this there is no definite reason preventing PSA technology from being established successfully in South Africa. The developments in technology for manufacturing molecular sieve has been very significant over the last 20 years and this will play a great role in further development of oxygen PSA systems. The various process plant manufacturing companies and plant technology discussed in great detail in sections 2.3 and 4.2 show that sufficient worldwide technology is readily available for implementation in South Africa.

5.1.2 Japanese Technology

As a result of the full technical and commercial evaluations undertaken on various forms of Japanese technology, it appears to be not the most suitable technology for the South African industrial requirements, due to the high capital costs and low power costs. Japanese technology should be reviewed from time to time to establish its relevance to South African applications and evaluate any major technological developments that may occur. Showa Engineering appears to be the most favourable Japanese company with which could be represented in South Africa. After visiting Showa Engineering in Tokyo their commitment to an African country is still not altogether clear to the author.

5.1.3 BOC Group Technology

The BOC group Centre of Excellence is committed to oxygen PSA progress and as a result are the most favoured of all PSA suppliers that were reviewed. BOC is currently rated among the top five PSA plant suppliers and with the current input into PSA technology, BOC is expected to lead world technological developments in the field of oxygen PSA plants.

BOC needs support from its group operating companies to test and evaluate their PSA knowledge. This will increase the group plant reference lists and enhance group understanding of PSA processes. BOC is close to first world technology. Any potential PSA oxygen application will be recommended to follow the BOC technology route that has been proved with the successful commissioning and evaluation of the pilot plant discussed in section 4.3.

5.1.4 PSA plant layouts

From the discussions in section 4.2.5.1 and Figure 4.3 it can clearly be seen that for any oxygen system requiring product oxygen at a capacity between 3 TPD and 100 TPD plants utilising adsorption techniques can supply this gas at a lower cost than the traditional methods of liquid oxygen from an on site cryogenic plant. The particular PSA plant layout shall be sized according to the customers requirements and constructed accordingly.

Technology is expanding at a very rapid rate and as a result the economic swing over from two bed PSA plants to three bed VSA plants is expected to change as time progresses. In November 1991, when this project was first conceived, it was thought that the cut-off between the two bed and three bed plants was near 8 TPD. For South African conditions of high capital costs and low power costs, this figure could easily increase to 40 TPD in the next few years, depending on the inflation rate and process plant developments.

5.1.5 Technology applicable to South Africa

Having conducted a total review of all the available PSA technology available to South Africa the Author is in a very strong position to make sound technological recommendations to the sponsor of this thesis on which strategic business decisions can be made.

Afrox Limited should keep within the BOC group technology field. In doing so, Afrox would be contributing directly to its parent company efforts, which will lead to a larger number of reference plants and will enhance the BOC group PSA understanding.

From the results of this study the BOC group standard plant design appears to be overall the most cost effective in terms of plant proposed to any potential gas requirement and financial situation. This could be either the two bed PSA type plant or the three bed VSA type plant.

The three bed VSA pilot plant must be used as a technology demonstrator and all results obtained from the plant should be kept confidential and used strictly within the BOC international group. This will enhance local engineering ability and serve as a reference to adsorption separation technology in South Africa and enhance Afrox's strategic business in the supply of oxygen from PSA systems.

Two bed plants very clearly have their use in South Africa. These would be suitable for many small applications such as Goldox, where gold leaching in cyanide is enhanced with oxygen enrichment or for a supply in a remote lying region where transport of liquid oxygen is expensive.

Three bed plants would be used where a two bed PSA plant is not economical or where a larger supply is required. This is usually for oxygen supply schemes to furnace steel mills and other large industrial applications, or to supplement the output from an existing ASU where a higher plant output capacity is required. Three bed plants may also be supplied in certain areas where there is a mining tax benefit whereby capital costs are written off during the first year of operation and operating and power costs are of extreme importance.

5.2 PSA PLANTS

5.2.1 *Plant design*

PSA plant process designs can be purchased from several process plant manufacturing companies. These designs may still be theoretical at this stage as very few PSA plants have been built to date. From the calculations made in section 3.3 it can be seen that the assumptions were not always as accurate as they could have been, but they allowed for very close estimation of the plant operating characteristics and the accurate equipment selection. The results obtained from the pilot plant show the plant operating characteristics for an adsorption separation plant are now known on which future plant designs and calculations can be based.

5.2.2 Plant construction

From the test results, as discussed in section 4.3.3, future plant construction will proceed with fewer problems since a large amount of potential problem areas have been identified. The recommendations for future plant construction mentioned in section 4.3.3 should be adhered to in order to make significant technological advances in this mode of technology. Plants can be constructed to very high manufacturing standards and as a result are commissioned with a minimum effort. The pilot plant demonstrated the capabilities of a typical VSA plant system. The successful commissioning showed that this plant can perform at the design conditions and that large technological developments in terms of understanding adsorption technologies have been made.

5.3 CONCLUSION

PSA plants can be locally designed, constructed and commissioned and are of an economical benefit to South African industry. It is expected that these plants will soon be marketed to many potential new oxygen applications. PSA plants are not expected to replace existing oxygen supply systems since they are more viable on a new and undeveloped site, where an existing TPV system or cryogenic plant would not have to be removed.

Having completed this study, the Author is now in a position to offer the South African industrial sector an ongoing support and service in the field of oxygen PSA technology by keeping up-to-date with international technology trends.

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APPENDIX

APPENDIX 1

PILOT PLANT COST BREAKDOWN

This Appendix shows the original budget that was justified to be spent on the construction and performance testing of the pilot plant mentioned in this text. It is important to note that during a project of this nature it is very difficult to justify all expenditure before a full design is undertaken and as a result, several changes to this cost breakdown were made during the project execution. The project was finally completed within the allowed budget.

Item	Comment	Cost (Rand)
Vessels and pipework		
Adsorber vessels	IR&D manufacture	248000
O_2 buffer vessel	IR&D manufacture	68000
Pipework material	Stockwell Tube	59980
Pipework Pre-fabrication	PFI	67020
Hydrostatic tests	Included IR&D	
10% non destructive test	Included IR&D	
O_2 cleaning	0.2 % of pipe costs	1200
Pipe Supports	Stockwell Tube	16400
Wedgewire screen	Wedgewire	15290
Flow distributers	Included IR&D	
TOTAL		475890
Air blower		
Machine and equipment	Aerzen with motor	175622
Performance test	3 days	2520
TOTAL		178142
Vacuum pump		
Machine and equipment	Hibon 2 stage Roots type	497680
Performance test	5 days	5040
TOTAL		502720
Valves		
18 Process valves	Fisher with actuators	244668
8 Throttle valves	Amri Gamma	9815
4 Non return valves	Mission Duo	12896
Drain valves	Worcester Audco	1275
Safety valves	Birkert	37172
Isolation Valves	Worcester Audco	2652
Pressure regulators	Mc Niel Steel	2500
Pilot valves	Worcester Audco	475
Valve gaskets	Mc Niel Steel	320
TOTAL		311773

Molecular sieves		
VSA II	UOP	258800
Activated alumina	Rhone Poulenc	22700
Ceramic balls	Chemplast	12139
TOTAL		293639
Instrumentation		
Pressure switches	Smar	37700
Thermocouples	RTD	2200
PLC Equipment	Texas Instruments	14000
O_2 Analyzer	Servomex	42180
Flow metering	Annubars	42000
Installation	Set Point	13500
Control panels	A C Controls	6300
Annunciators	Conlog Omni 16	5470
TOTAL		163350
Electrical		
HV Distribution	Components	40000
LV Distribution	Components	74392
Control panels	A C Controls	12500
Transformers	Cawse & Malcolm	10000
Sundry components		2500
Lighting		3000
Cables	Stateway Electrical	40000
Lightning protection		7500
Inspection and testing	Test a Relay	5000
Installation	T J Electrical	22000
TOTAL		216892
Civils		
Equipment foundations	AT&E	24580
Fences and gates	Rand Fencing	10000
Control cabin	Portacabin	23500
Roofing	Chromadeck	24000
TOTAL		82080
Services		
Instrument air	BOGE Screw compressor	17500
Cooling tower	Sulzer	22200
Water reticulation	Stockwell Tube	4500
Installation	Contract	4000
TOTAL		48200
Transport		
Road Transport	Jumbo Machine Movers	12500
TOTAL		12500
Erection		
Air blower	Airgas Compressors	2000

Vacuum pump	Torrvac	4000
Adsorber vessels	IR&D	30000
Buffer vessel	IR&D	10000
Pipework supports	PFI	20000
Crane hire	Johnssons Crane	45000
Scaffolding	Hire costs	5000
Painting	Kelly	15000
Fire protection	Chubb Fire	2500
Consumables	General	8500
TOTAL		142000

Site running costs

Telephone		6000
Vehicle	Site Vehicle	5000
Site meals		5000
Expense claims		5000
TOTAL		21000

Commissioning and training

Leak testing		30000
Machinery		50000
Vessel Filling	Filling equipment	42000
Process		58000
Training		20000
Overseas vendors		56000
Local contractors		20000
Extras		10000
Travel	Afrox Engineer in Japan	30000
TOTAL		316000

Management Fees

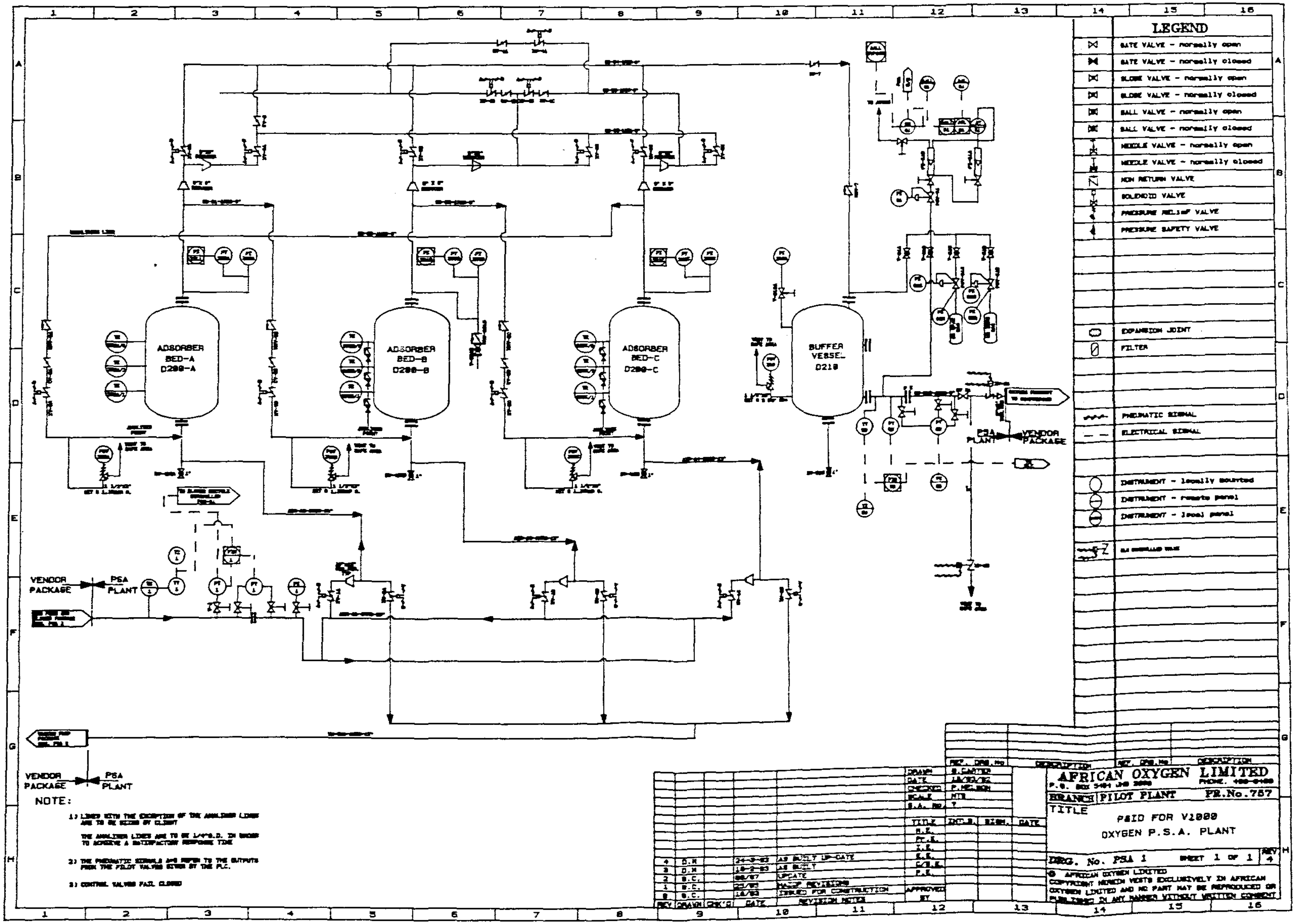
Process	45000
Instrumentation	38000
Electrical	33000
Civils	30000
Mechanicals	46000
Drawing Office	40000
Site Foreman	84000
TOTAL	316000

PROJECT TOTAL	3080186
SPARES (3 %)	92406
ESCALATION (6 %)	184811
GRAND PROJECT TOTAL	3357403

Note: Escalation has been calculated on local and overseas inflation rates during the project period.

APPENDIX 2

PILOT PLANT P&ID



NOTE:

1) LINES WITH THE EXCEPTION OF THE ANALISER LINES ARE TO BE SIZED BY CLIENT

THE ANALISER LINES ARE TO BE 1/4"O.D. IN ORDER TO ACHIEVE A SATISFACTORY RESPONSE TIME

2) THE PNEUMATIC SIGNALS A-G REFER TO THE OUTPUTS FROM THE PILOT VALVES GIVEN BY THE PLC.

3) CONTROL VALVES FAIL CLOSED

REV	DRAWN	CHK'D	DATE	REVISION NOTES
4	D.M		24-3-93	AS BUILT UP-DATE
3	D.M		18-2-93	AS BUILT
2	B.C.		08/87	UPDATE
1	B.C.		25/85	MAJOR REVISIONS
0	B.C.		14/83	ISSUED FOR CONSTRUCTION

DRAWN	B.CARTER
DATE	[illegible]
CHECKED	P.NELSON
SCALE	NTS
S.A. No.	?

TITLE	INIT.	SIGN.	DATE
R.E.			
Pr.E.			
I.E.			
E.E.			
C/S.E.			
P.E.			
APPROVED BY			

AFRICAN OXYGEN LIMITED
P.O. BOX 5404 JHB 2000 PHONE. 490-0400

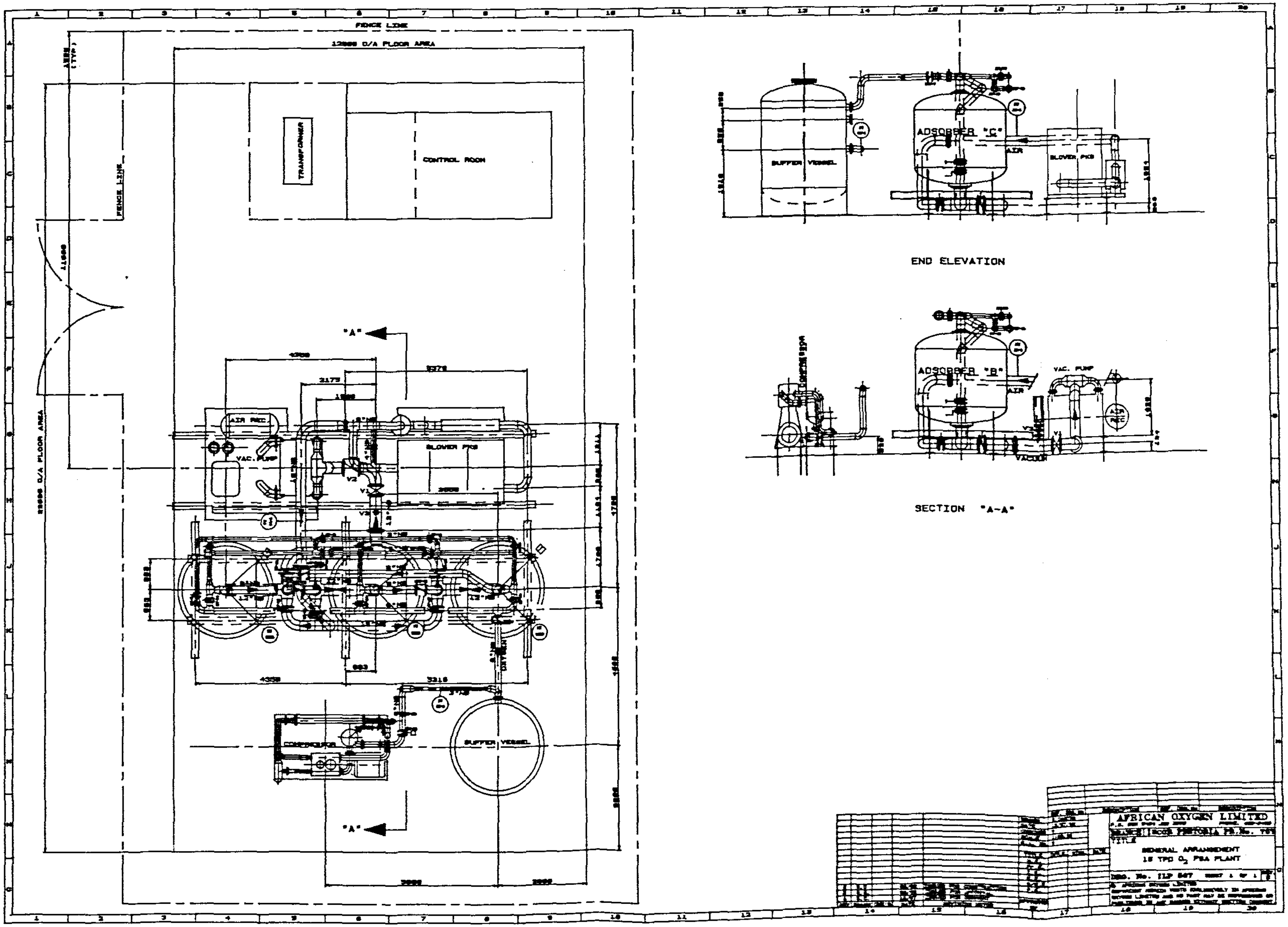
BRANCH PILOT PLANT PR.No.757

TITLE
P&ID FOR V1000
OXYGEN P.S.A. PLANT

DRG. No. PSA 1 SHEET 1 OF 1 REV 4

APPENDIX 3

GENERAL ARRANGEMENT DRAWING


FENCE LINE
12000 O/A FLOOR AREA
TRANSFORMER
CONTROL ROOM
FENCE LINE
11000
25000 O/A FLOOR AREA
"A"
4300
2175
3375
AIR REC.
VAC. PUMP
BLOWER PKG
V2
V1
V3
3000
4350
3315
COMPRESSOR
BUFFER VESSEL
"A"
3000
3000
BUFFER VESSEL
ADSORBER "C"
AIR
BLOWER PKG
END ELEVATION
COMPRESSOR
ADSORBER "B"
AIR
VAC. PUMP
AIR REC.
VACUUM
SECTION "A-A"
AFRICAN OXYGEN LIMITED
PRETORIA
TITLE
GENERAL ARRANGEMENT
15 TPD O₂ PSA PLANT
DRG. No. ILP 507

APPENDIX 4

CONFIDENTIALITY AGREEMENT

CONFIDENTIALITY AGREEMENT

between

AFRICAN OXYGEN LIMITED
("Afrox")

and

PETER ROBERT NELSON
("Nelson")

and

THE CAPE TECHNIKON -
MECHANICAL ENGINEERING DEPARTMENT
("the Technikon")

1. BACKGROUND

1.1 During January 1992 Nelson will be submitting an Oxygen From Air By Pressure Swing Adsorption ("PSA") project proposal to the Technikon with a view to presenting a full PSA project ("the PSA Project") to the Technikon for adjudication and the award of a Masters Diploma in Technology.

1.2 The PSA project proposal and the PSA Project will both embody certain of Afrox's confidential strategies, ideas, technical know-how and documents ("the Confidential Material") and Afrox therefore requires Nelson and the Technikon to respect the secrecy of the Confidential Material.

1.3 The parties have accordingly agreed to enter into this Confidentiality Agreement on the following terms and conditions,

2. CONFIDENTIALITY

2.1 In consideration of the Confidential Material that Afrox will make available to Nelson for the PSA project proposal and the PSA Project, Nelson and the Technikon undertake that they will -

2.1.1 treat the Confidential Material as secret and confidential;

2.1.2 not use or cause the Confidential Material to be used other than for Nelson's PSA project proposal and the PSA Project and the adjudication thereof for his Masters Diploma in Technology;

2.1.3 not disclose or cause the Confidential Material to be disclosed to any party other than to those members of the Technikon's staff who have a direct interest therein.

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2.2 The restrictions imposed on Nelson and the Technikon under Clause 2.1 above may be relaxed with the prior written approval of Afrox and at Afrox's sole discretion should Nelson or the Technikon wish to use any of the Confidential Material for teaching, for education or generally for advancing engineering and technical knowledge in winning oxygen from air by pressure swing adsorption.

3. LIMITATION OF RIGHTS

No intellectual property rights or any other rights or licences in and to the Confidential Material are intended to be conveyed by Afrox to Nelson or the Technikon other than to embody the same in the PSA project proposal and the PSA Project.

4. NON-APPLICABILITY OF THIS AGREEMENT

4.1 Notwithstanding the provisions of Clauses 2 and 3 above, this Confidentiality Agreement shall not apply to any of the Confidential Material which Nelson or the Technikon -

4.1.1 can prove to have been in their possession or within their knowledge at the date when the same is made available by Afrox for the PSA project proposal or for the PSA project;

4.1.2 can demonstrate is public knowledge;

4.1.3 obtains from a third party who has no duty of secrecy or confidentiality to Afrox.

5. DURATION OF THIS AGREEMENT

This Confidentiality Agreement shall be deemed to have come into effect on 1 November 1991 and shall remain in force until 31 October 2006.

Signed at JOHANNESBURG on this 13th day of January 1992

for Afrox
being duly authorised

Signed at JOHANNESBURG on this 13th day of January 1992

Peter Nelson

Nelson

Signed at CAPE TOWN on this 24.. day of January 1992

for the Technikon
being duly authorised

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