# CHAMBER OF MINES RESEARCH ORGANIZATION

# Report to the

## WATER RESEARCH COMMISSION

on

# THE EVALUATION OF SLURRY REVERSE OSMOSIS FOR

# THE DESALINATION OF CALCIUM SULPHATE

## SCALING MINE SERVICE WATER

by

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#### EVALUATION OF SLURRY REVERSE OSMOSIS FOR THE DESALINATION OF CALCIUM SULPHATE SCALING MINE SERVICE WATER

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EVALUATION OF SLURRY REVERSE OSMOSIS FOR THE DESALINATION OF CALCIUM SULPHATE SCALING MINE WATER

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#### PREFACE

The gold mining industry buys approximately 500 Mt/day of water from Water Boards for consumption within mines. Because of the ever increasing demands on South Africa's limited water resources and the gradually deteriorating water quality, the cost of high quality Water Board water is increasing steadily.

The use of water for mining results in significant increases in the salt content of the water, making it more corrosive and scale-forming and this in turn increases the maintenance costs of mine water reticulation systems. The most recent estimate for the cost penalty of poor quality service water in the gold mining industry is R300 million per annum. To limit the build-up of salts, up to 440 M<sup>2</sup> per day of saline water is discharged from the mines. Some of this water may enter the surface or ground water environment and this practice is becoming increasingly unacceptable to the water authorities and public environmental lobby.

Reverse osmosis has been identified as a suitable technology for the desalination of mine service water. Mine waters may be classified into two categories, namely those which have a scaling potential with respect to calcium sulphate and those which do not. COMRO has, in 1986/87, carried out definitive research and shown that conventional tubular reverse osmosis is viable for the desalination of non-scaling brackish mine water.

This report describes the test work carried out on two alternative Slurry Reverse Osmosis pilot plants treating mine service water at the Hercules shaft of the ERPM gold mine at Boksburg. The mine water at ERPM is classified as "scaling" and it has a high calcium sulphate content. It is shown that, with appropriate pre-treatment, the Slurry Reverse Osmosis plant process is technically and economically viable on this type of mine water and gives a high quality product.

The work is considered definitive and no further test work on the slurry reverse osmosis process using this type of feed water is required. Sufficient data are now available to permit the design of a large scale process. A design and cost estimate has been prepared for a plant to produce  $5,78 \ l/s$  (500 kl/day) of desalinated mine service water at a recovery of 90 per cent and is available on request as a separate report.

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#### <u>SUMMARY</u>

#### INTRODUCTION

The gold mining industry uses large quantities of water, notably for cooling and dust suppression in deep mining and stoping. Mine service water quality varies significantly from mine to mine, but has generally deteriorated since the Water Boards introduced restrictions during the droughts in the 1980's causing the mines to place more emphasis on recycling and reuse of the water. The latter in turn, has led to an increase in the corrosion and scaling potential of the mine waters.

Although there are no exact figures on the cost of poor quality service water in mines, the most recent estimate is up to R300 million per annum for the gold mining industry as a whole. Recent developments such as the use of hydro-power, as well as possible future implementation of more restrictive legislation on the discharge of waste waters, have highlighted the poor quality of mine service waters currently in use in reticulation systems, and the need for improving their quality in order that they can be reused or safely disposed of.

Mine waters can be broadly classified into two categories, namely those which have a scaling potential with respect to calcium sulphate and those which do not. Approximately 75 per cent of mine service waters are classified as "scaling" and 25 per cent as "non-scaling".

Previous work by COMRO (1987) has demonstrated that conventional reverse osmosis is a viable process for the desalination of "non-scaling" mine water containing a high concentration of sodium chloride.

The slurry reverse osmosis (SPARRO) process is one of the most promising technologies available for the desalination of calcium sulphate scaling mine waters. Previous pilot plant tests carried out by COMRO at ERPM gold mine (1984) have confirmed the technical viability of the process.

The aims of the study were to establish whether or not the seeded slurry process was a technically viable process for the full scale desalination of calcium sulphate scaling mine waters; to establish the product water recovery and salt rejection levels that could be achieved by the process; to establish those pre-treatment techniques which would be required to ensure good plant performance; to establish whether membrane fouling/scaling would occur while treating a mine water with a high calcium sulphate concentration; to evaluate and compare the alternative design features of two variants of the process; to obtain detailed design data; to establish the operating requirements; to prepare a design for a larger plant; and to estimate the capital and operating costs for a plant capable of producing 5,78 l/s of desalinated mine service water.

#### FINDINGS AND DISCUSSIONS

The SPARRO (Slurry Precipitation and Recycle Reverse Osmosis) and MLT (Membrane Lifetime Test) and the common pre-treatment plant were operated on a continuous basis (24 hours per day), treating scaling mine water from the ERPM gold mine at Boksburg. The MLT plant was similar to that used in the previous COMRO work, while the SPARRO plant incorporated certain novel features. The units were operated from February 1989 to August 1990 for 8 549 hours (MLT) and 5 983 hours (SPARRO).

During 1989 the pre-treatment plant was designed and operated to remove iron and manganese hydroxides which may cause membrane degradation resulting in low salt rejections and low flux. This was accomplished by applying the following pre-treatment: raising the pH to 10 by the addition of caustic soda solution; oxidation of iron and manganese using potassium permanganate; removal of suspended solids and the products of oxidation by flocculation; and then filtration through dual media (sand/anthracite) filters. This special pre-treatment was followed by standard water conditioning for cellulose acetate membranes, i.e. pH adjustment to between five and six, and temperature control to 25 °C.

The performance of both the MLT and SPARRO plants during 1989 was unsatisfactory with membrane degradation which caused a drop in the salt rejection and increased flux. The SPARRO plant had additional problems which included restricted output from the slurry feed pump, difficulties in controlling reactor level, and blockages of the hydrocyclone purge.

The pre-treatment system was modified in December 1989 by discontinuing both the initial pH adjustment to 10 and the addition of potassium permanganate. This meant that no iron and manganese removal was taking place. Chlorination was introduced in November 1989. Operations on both plants were successful during 1990 and all major process problems were overcome. The performance of the membranes on the MLT plant was satisfactory and complied with the manufacturer's design data. The performance of the membranes on the SPARRO plant, though much improved, was inferior to the MLT plant. The SPARRO membranes required cleaning and restoration.

The single slurry feed pump system was found to be superior to the twin pump system in the SPARRO plant in terms of improved reactor level control and increased module life.

Typical values of raw mine water feed and desalinated product water quality are shown in Table 1.

Determinand	t	Raw Mine	MLT Product	SPARRO Product
рH		4,63	5,1	5,09
Conductivity at 25 °C	(mS/m)	206	55	82
Suspended solids	(mg/ <i>l</i> )	54	0	0
Total dissolved solids (TDS)	(mg/ <i>l</i> )	2124	388	561
Ca <sup>2+</sup>	(mg/\$)	361	14	17
Na +	(mg/ <i>l</i> )	121	70	102
SO4 <sup>2-</sup>	(mg/ <i>l</i> )	1211	147	244
Ct-	(mg/ <i>l</i> )	50	41	48

#### 

\* 21st March 1990.

As indicated by Table 1, the product water from the MLT plant was of a high quality (conductivity 72 mS/m) and would be suitable for reuse after a slight upward pH adjustment. The product water is compared in Table 2 with that required for effluent discharge and the SABS maximum allowable limits for drinking water.

Determinand	SPARRO Product Water	MLT Product Water	General Effluent Discharge Standards	SABS Maximum Allowable Limit
pH Conductivity at 25 °C (mS/m)	5,09 82	5,1 55	5,5 - 9,5 Not to be increased by more than 75 mS/m above that of the intake water, i.e. 155 max*	5,5 - 9,5 300
Socium (mg/x)	102	70	more than 90 mg/ $l$ above that of the intake water, i.e. 106 max*	400
Manganese (mg/l)	2,1	1,0	0,4	0,1
Chloride (mg/l)	48	41	Not specified	600
lron (mg/l)	0,1	0,6	Not specified	1,0
Sulphate (mg/l)	244	147	Not specified	600

# Table 2 COMPARISON OF PRODUCT WATER DETERMINANDS WITH STANDARD EFFLUENT DISCHARGE AND DRINKING WATER REQUIREMENTS

\* Intake water refers to water supplied by the Water Board

With regard to discharging the product water to the environment, a slight upward adjustment of pH is required with further treatment to reduce manganese levels. For human consumption of the product water, a slight upward adjustment of pH and a further reduction of manganese is required to meet the SABS maximum allowable limits for chemical quality (health related parameters were not considered in this study).

Operating costs are estimated to be R1,34 c/kl (1990). The costs are made up of the following: membrane replacement (46 %); electricity consumption (34 %); pump maintenance (17 %); pre-treatment (3 %). The membrane cost is based on an estimated lifetime of two years.

The capital cost of a slurry reverse osmosis plant with a capacity of 5,78 l/s (500 kl/day) of product is estimated to be R2,58 million in 1990 terms. The detailed design and cost estimate are presented in a separate report which is available on request.

#### MAIN CONCLUSIONS

- 1. The seeded slurry reverse osmosis process was demonstrated to be capable of successfully desalinating calcium sulphate scaling mine water.
- 2. A module lifetime of at least two years can be achieved if the specified pre-treatment and operating procedures are followed.
- 3. The single slurry pump system as used in the MLT plant is superior to the twin pump system as used in the SPARRO plant in terms of reactor level control and module performance.
- 4. It is not necessary to remove iron and manganese in the pre-treatment stage.
- 5. The capital cost of a plant producing 5,78 l/s (500 kl/day) of desalinated, scaling mine water is estimated to be R2,58 million in 1990 terms.
- 6. The operating costs of a slurry reverse osmosis plant operating on scaling mine water are estimated to be R1,34/k<sup>l</sup> in 1990 terms.
- 7. The product water produced by the plant was suitable for use in hydro-power systems, for human consumption or for effluent discharge after minor adjustments of certain water quality determinands. For continuous use as domestic water, further consideration would have to be given to health related water quality parameters.
- It is felt that further pilot plant evaluation of the process in similar scaling water is not required. Data obtained from this study are sufficient to enable realistic operating cost estimates to be made for full scale plants.

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#### 1 INTRODUCTION

The South African gold mining industry circulates 5 500 M<sup>*l*</sup>/day of water for mining operations<sup>(1)</sup>. Water is used for many purposes underground including drilling, dust suppression, sweeping, washing and cooling. The volumes of water recirculated and reused on mines has increased in recent years owing to restrictions imposed by the various Water Boards especially during droughts. Increased recirculation has led to a general increase in the dissolved solids content of the mine service waters, making them potentially more scaling and possibly more corrosive.

The main reasons for considering desalination in the gold mining industry include the following:

- improving the quality of water in order that it can be cost-effectively reused as service water.
- . improving the quality of water for use in hydro-powered mining systems where the hydrostatic head of water is used to power machinery underground<sup>(2, 3)</sup>
- . producing potable water as an alternative to purchasing board water.
- . producing potable water in emergencies when board water is unavailable due to drought
- . protecting water reticulation systems against corrosion and scale formation.
- . improving the quality of water to be discharged to the environment.

Mine waters can be broadly classified into two distinct groups, namely those that have a scaling potential with respect to calcium sulphate and those that do not. The latter are essentially sodium chloride waters and comprise only about 25 per cent of all mine service waters and are found predominantly in the Orange Free State gold fields. Previous work carried out by COMRO at the Beatrix Gold Mining Co. Ltd in 1987 showed that, with appropriate pretreatment, the conventional tubular reverse osmosis process operates well on non-scaling water<sup>(4)</sup>. Since calcium sulphate is a sparingly soluble salt  $(2,23 \text{ g/l} \text{ at } 0^{\circ} \text{ C})$ , high calcium or sulphate concentration in mine water can lead to scaling problems. Data gathered from two extensive surveys of the service water in gold mines have revealed that about 75 per cent of them would become scaling when concentrated in a desalination process operating at 80 per cent water recovery (5, 6). At the ERPM gold mine near Boksburg, the mine service water is brackish and is almost saturated with calcium sulphate. Conventional reverse osmosis and electrodialysis reversal processes are not suitable for use with water of this quality. The calcium sulphate slurry precipitation and recycle reverse osmosis (SPARRO) process which has been developed by COMRO has previously been shown in principle<sup>(7)</sup> and on a small scale to be suitable for the desalination of this type of scaling mine water. Two plants, namely the

 $0,7 \ l/s$  SPARRO plant and the  $0,17 \ l/s$  Membrane Lifetime Test (MLT) plant, were installed at the ERPM gold mine to evaluate, optimize and develop the process so that it may be applied commercially on a larger scale.

This report describes the design changes, results and experience obtained during the operation of the two desalination pilot plants between February 1989 and August 1990. The common pretreatment section was also extensively modified and optimized during this period, and together with the desalination sections are evaluated in this report. A design and capital costing has been prepared for a larger plant with a capacity of 5,78  $\ell$ /s (500 k $\ell$ /day) and is available as a separate report.

#### 2 <u>OBJECTIVES</u>

- 1. To develop and define the pretreatment process equipment and operating conditions required for the optimum operation of a slurry reverse osmosis desalination system.
- 2. To establish the economic lifetime of tubular cellulose acetate membrane modules for desalinating scaling mine water in a seeded slurry mode.
- 3. To develop and evaluate the performance and maintenance requirements of the NATIONAL and CROWN high pressure slurry feed pumps.
- 4. To compare the twin high pressure feed pump system used in the SPARRO plant with the single pump system in the MLT plant.
- 5. To evaluate the effect of contact time between pretreated water and calcium sulphate slurry on the performance of membrane modules.
- 6. To evaluate the performance of the "tapered" versus the "straightthrough" module stack.
- 7. To develop and evaluate the hydrocyclone system to control purge and suspended solids concentration.
- 8. To develop and optimize operating and maintenance procedures.
- 9. To establish operating costs.
- 10. To establish design data to be used in the design of a larger commercial plant.
- 11. To prepare a design and estimate the capital cost of a pretreatment and slurry reverse osmosis plant to produce 5,78 l/s (500 kl/d) of desalinated mine water.
- 12. To promote the SPARRO process and seek applications within the mining industry.

#### 3 <u>SLURRY REVERSE OSMOSIS TECHNOLOGY BACKGROUND</u>

### 3.1 Principles of Osmosis and Reverse Osmosis

Osmosis is a natural phenomenon involving fluid flow across a semi-permeable membrane. Such membranes are selective in that certain components of a solution, usually the solvent, can pass through them while others, usually the dissolved solids, are retained<sup>(8)</sup>. In osmosis, water molecules from pure water will pass through a semi-permeable membrane into salty water in order to equalize the salt concentrations on both sides of the membrane, as shown in Figure 1(a). Figure 1(b) shows the dynamic equilibrium established by the movement of the water; water from the pure water source is still passing into the salty water but the pressure differential between the two solutions, brought about by the change in volume, is causing water molecules to flow out of the salty source at the same rate. The differential pressure is known as the osmotic pressure, and it is defined as the externally applied pressure to the salty solution side necessary to establish equilibrium between the two solutions. If the external pressure applied to the salty water side is greater than the osmotic pressure, then water molecules will pass from the salty solution into the pure water side. This phenomenon is known as reverse osmosis (RO) and is illustrated in Figure 2.



(a) Osmosis

(b) Dynamic equilibrium

#### Figure 1 PRINCIPLE OF OSMOSIS



#### Figure 2 PRINCIPLE OF RO FOR DESALINATION

In the early 1960s, the development of the asymmetric cellulose acetate membrane permitted RO to become the successful practical process it is today. Currently, there are three major membranes on the market: cellulose acetate, aromatic polyamide polymer, and thin-film composite. The latter two membranes can tolerate wide pH variation (pH 4-11) and are resistant to hydrolysis, but are very sensitive to chlorine degradation. Cellulose acetate membranes, on the other hand, can tolerate chlorine levels of 0,5 to 1,0 mg/ continuously and 10 mg/ as a shock dose. They are usually used in conjunction with chlorination to prevent biological attack of the membrane, to which they are susceptible. Cellulose acetate hydrolyses at high and low pH, and high temperature, as shown in Figure 3. For this reason the feed water temperature has to be controlled to less than 30 °C, and the pH to between 4,0 and 6,0.



# Figure 3 THE EFFECT OF TEMPERATURE AND pH ON THE HYDROLYSIS OF CELLULOSE ACETATE

Reverse osmosis membranes have been fabricated in three configurations: namely spiral-wound, hollow-fibre and tubular. As the name suggests, tubular reverse osmosis (TRO) membranes are in the form of tubes. The membrane material itself is coated onto the inside surface of a synthetic porous tube supported to withstand the high operating pressure required for the process (4 000 kPa).

The tubular membrane configuration offers certain advantages over the more conventional membrane arrangements of spiral-wound and hollow-fibre. It can tolerate feed waters with suspended solids, and the tubes facilitate easy cleaning of the membrane, both chemically and mechanically (sponge-ball cleaning). Effectively, this means that they can tolerate a lower degree of feed pretreatment in terms of suspended solids removal, compared to both hollow-fibre and spiral wound which require a high degree of suspended solids removal (i.e. sand filtration followed by cartridge filtration at micron level), which adds to the overall operating cost. Hollow-fibre membranes, and to a certain extent spiral-wound membranes, are difficult to clean chemically and nearly impossible to clean mechanically<sup>(9)</sup>, particularly <u>in situ</u>. Tubular reverse osmosis membrane modules are manufactured in South Africa.

#### 3.2 Principles of Slurry Reverse Osmosis

Conventional reverse osmosis (RO) systems, whether they are of spiral, hollow fibre, or tubular configuration, require feedwaters free from fouling and scaling components. In practice this requirement dictates that the feedwater be free of suspended solids, oils, grease, and colloidal materials such as iron and manganese. Further, the water recovery must be such that the solubility limit of the dissolved salts in the feedwater is not exceeded, in order to preclude the formation of scale on the membrane surfaces.

To date, most applications of reverse osmosis, and membrane desalination processes in general, have been limited to the desalination of relatively uncontaminated feed streams, such as brackish ground and surface waters or sea water. In most of these applications, the production of pure water was the major objective and hence the water recovery was not of prime importance.

Where RO has been used in waste water applications, "pretreatment" of the water feed to the plants has been a major consideration and important cost factor in the design and operation of the system. In many cases the pretreatment requirements have made the use of conventional RO membrane processes uneconomic. Gold mine service waters may be included in this category as they have relatively high concentrations of suspended solids, aluminium, iron, manganese, and silica, as well as significant amounts of oil and grease. In addition, they are in many cases nearly saturated with calcium sulphate, the principal factor which limits the water recovery of the system. For conventional membrane processes to work effectively on these waters, an extensive pretreatment system would be necessary. Suspended solids removal, reduction in iron and manganese concentration, and oil and grease removal would be required. In addition, to obtain a reasonable level of water recovery, chemical softening, using lime and soda ash,

would be required to precipitate calcium and magnesium and reduce silica levels. Pretreatment costs would thus tend to make the treatment of mine service waters by conventional reverse osmosis uneconomic.

The slurry reverse osmosis process was developed to overcome many of the difficulties encountered in the application of conventional RO processes to waste water treatment. The preferential seeding process is illustrated in Figure 4. In the simplest terms, a slurry of "seed" crystals, principally composed of calcium sulphate, is incorporated into the feed water of a tubular reverse osmosis system. These seed crystals then serve as preferential sites for the growth of additional crystals of calcium sulphate, silica and other salts and prevent the formation of scale on the surface of the membrane.

Figure 4 also shows that seed material is recycled from the concentrate to the feed. This seed material is added to the system at start-up. After the initial load is added, no further outside source of seed material is required as the process is self sustaining through the continuous crystallization of calcium sulphate. It should be noted that the total dissolved solids (TDS) of the seed slurry feed to the RO modules is much higher than the mine water feed TDS (as shown by the typical TDS values in Figure 4) because of the calcium sulphate recycle system.

Tubular reverse osmosis systems are required for the SRO process as slurries of 20 000 to 40 000 mg/<sup>g</sup> solids are circulated continuously in the system. Such concentrations of suspended solids would rapidly result in complete blockage of either spiral or hollow fibre configuration RO modules. These high concentrations of suspended solids are achieved through the use of a hydrocyclone in the concentrate return stream. The fraction of the hydrocyclone-processed stream that is enriched in seed material is returned to the seed recycle tank: the seed depleted stream then becomes the waste and is purged to control the salinity of the system and the overall water recovery. This recycle of seed, plus the fact that SRO systems operate at high water recovery, results in high total suspended solids levels in an operating SRO system. The high recoveries can be achieved on waters saturated with calcium sulphate without producing excessively high osmotic pressures because the calcium sulphate content of the feedwater is precipitated in the seed slurry process.



# Figure 4 SEEDED SLURRY PRECIPITATION REVERSE OSMOSIS PROCESS

#### 3.3 Previous Research By COMRO (1983 - 1987)

A feasibility study initiated by COMRO and prepared by Resources Conservation Company International (RCCI)<sup>(5)</sup> indicated that from technical, practical and economic considerations, the seeded reverse osmosis (SRO) process represents one of the most promising technologies available for the desalination of calcium sulphate scaling mine waters. Small-scale pilot plant tests (0,05  $\ell$ /s) were undertaken with the process at the East Rand Proprietary Mines (ERPM) by COMRO in 1984 in collaboration with RCCI and the Water Research Commission (WRC). These tests confirmed the technical viability of the process as applied to CaSO<sub>4</sub> scaling-type mine service waters<sup>(7)</sup>.

Based on the promising small-scale pilot plant results and the potential wide ranging application possibilities for the process in the South African mining industry, the process was tested on a larger scale. COMRO operated a 0,5 l/s pilot plant supplied by RCCI at ERPM in 1986. Numerous problems were experienced with this unit, with only 600 hours of operation being possible during 1986. The major problems were with the high pressure feed and interstage pumps and rapid failure of the membranes.

#### 3.4 Previous Research By Resources Conservation Company International (RCCI)

The research<sup>(10)</sup> was carried out over a ten month period by RCCI for the Electric Power Research Institute (EPRI) at an electricity generating station of the Utah Power and Light Company. Power plant waste water treatment systems rarely incorporate membrane processes because the membranes often experience fouling due to inorganic scaling. Although a properly designed and operated pretreatment system can avoid this fouling, the capital and operating costs for the entire system are usually prohibitive. The study included operation at 80 per cent water recovery on cooling tower blowdown, operation at 65 per cent on a flue gas desulphurization thickener overflow feed, and a comparative evaluation of five different tubular cellulose acetate membranes.

Although the membranes showed no signs of fouling from inorganic scaling, they deteriorated rapidly during the first two test phases. The rapid failure was attributed to the presence of hard sharp particles of fly ash. Economic analysis indicated that, under certain circumstances, SRO could be more cost-effective than traditional waste water treatment processes such as brine concentration or conventional reverse osmosis with pretreatment. However, the cost and assumed lifetime of the membranes greatly affect estimates of capital and operating costs. It was concluded that the resolution of membrane fouling and deterioration concerns would permit seeded reverse osmosis to become an important part of integrated water management schemes.

#### 3.5 <u>Patents</u>

Seeded slurry reverse osmosis was conceived and developed by RCCI, then further developed by COMRO. It is a proprietary process which allows high water recovery from scaling waters without the extensive pretreatment normally required for conventional

reverse osmosis systems. The RCCI process is protected by South African patent No. 85/0168 (dated 28 May 1986) which incorporates a previous USA patent No. 4207183 (dated 10 June 1980).

The inventions claimed by COMRO with reference to the SPARRO process are covered by South African patent application No. 89/3746 (dated 16 May 1990).

#### 3.6 Collaborative Agreements

A memorandum of agreement in connection with the development of seeded reverse osmosis technology was signed on 24 June 1988 between COMRO, WRC, ISCOR and MEMBRATEK.

The agreement allocates the rights in respect of inventions and developments as a result of the collaboration during the period January 1988 to August 1990.

#### 3.7 Licences

Under the terms of an agreement between RCCI and Membratek dated 11 September 1987, Membratek was appointed sole and exclusive licensor of RSA patent No. 85/0168 and all relevant proprietary information. The ownershipr of developments covered by RSA patent No. 89/3746 is shared by Membratek, COMRO and the Water Research Commission.

#### 4 DESCRIPTION OF THE PILOT PLANTS AT ERPM (1989/1990)

#### 4.1 <u>Pretreatment</u> (Figure 6)

The common pretreatment plant operates at up to 1,1 l/s to treat the raw mine service water from the ERPM gold mine so that it becomes suitable for the MLT and SPARRO plants which are situated downstream. Some of the equipment and operating procedures were changed between February and December 1989. The first system used during 1989 is shown in Figure 5 and the final modified system which was used in 1990 is shown in Figure 6. The final system is described below and the changes are covered in Section 6.3.

(a) Raw Mine Water Storage. Mine water is pumped to the pretreatment plant during the night shift and is stored in two tanks with capacities of 50 m<sup>3</sup> and 20 m<sup>3</sup> giving a



Figure 5 PROCESS FLOW DIAGRAM OF 1.1 <sup>1</sup>/s PRETREATMENT PLANT BEFORE MODIFICATION IN 1989 - FIRST SYSTEM



# Figure 6 PROCESS FLOW DIAGRAM OF 1.1 1/s (3.96 m<sup>3</sup>/h) PRETREATMENT PLANT AFTER MODIFICATION IN 1990 - FINAL SYSTEM

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total capacity of 70 m<sup>3</sup> and a reserve of 17 hours. The main 50 m<sup>3</sup> storage tank is made of epoxy painted carbon steel and the auxiliary 20 m<sup>3</sup> tank is constructed of stainless steel to prevent corrosion by the acid mine service water.

- (b) Pretreatment Tank. Raw mine water flows by gravity on demand from the storage tanks into the 1,5 m<sup>3</sup> HDPE stirred pretreatment tank. Sodium hypochlorite and polyelectrolyte are added from a multiple head dosing pump. Sludge is recycled from the base of the clarifier which is situated immediately downstream. The polyelectrolyte solution can be added to the discharge line from the clarifier feed pump to prevent floc breakdown.
- (c) Clarifier. Water is pumped from the pretreatment tank to the clarifier where the flocs develop and settle. The clarifier is made of GRP and has a diameter of 1 950 mm and a capacity of 6,3 m<sup>3</sup>. The nominal upflow rate at a throughput of 1,1 l/s is 1,3 m/h. The clarifier is fed from a central flocculating tube.
- (d) pH Adjustment Tank. Clarified water overflows at the periphery of the clarifier and runs by gravity to the pH adjustment tank. The tank, which is made of rubber lined carbon steel, is stirred and has a capacity of 2,1 m<sup>3</sup>. Provision is made for the pH to be controlled between pH 4-6 by the addition of caustic soda solution and sulphuric acid. This facility was not used from April to August 1990 as it was found that the pH of the water fed to the MLT and SPARRO plants was nearly always acceptable.
- (e) Sand Filters. Water from the pH adjustment tank is pumped through two downflow sand/anthracite filters installed in parallel. The filters are backwashed from the pH adjustment tank once per shift.
- (f) Pretreated Water Storage Tank. Filtered water is stored in a mild steel rubber lined tank with a capacity of 2,1 m<sup>3</sup>. The tank provides a buffer storage of approximately half an hour when both the MLT and SPARRO plants are running. Water is supplied to the MLT and SPARRO plants by a ring main. The point of delivery for the MLT plant is into the reactor. The point of delivery for the SPARRO plant is into the raw feed water tank upstream of the high pressure mine water (MONO) pump.

#### 4.2 <u>Membrane Lifetime Test (MLT) Plant</u> (Figure 7)

(a) Reactor. Pretreated water is pumped to the reactor on demand to maintain a constant level. The reactor which has a volume of 13 m<sup>3</sup> is made of GRP and is equipped with a central top mounted agitator. The reactor contains a suspension of fine calcium sulphate particles which are formed and grown by the crystallization of calcium sulphate from the supersaturated reject stream which is recirculated from the downstream membrane module bank. The residence time in the reactor is



Figure 7 PROCESS FLOW DIAGRAM OF 0.17 1/s (0.612 m<sup>3</sup>/h) MLT PLANT

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approximately four hours based on the rate at which the suspension is pumped from the bottom of the reactor to the membrane module bank. The target concentration of calcium sulphate is  $20\ 000 - 40\ 000\ mg/l$  and the minimum level is  $10\ 000\ mg/l$ .

- (b) Slurry Feed Pump. The saturated suspension from the reactor is delivered to the membrane bank by the slurry feed pump at a pressure of 3 000 kPa. The pump is a Crown Chrome Plating Co. triplex pump Model 707 with three 38 mm diameter chromium plated plungers and fitted with rubber chevron gland seals. The output of the pump is 0,83 l/s (3 m<sup>3</sup>/h) at 243 rev/min.
- (c) Membrane Module Bank. The bank is made up from 20 standard Membratek cellulose acetate tubular reverse osmosis modules. The modules are arranged with four passes in parallel and with five modules in series in each pass. The velocity of the suspension as it passes through the tubular membranes is approximately 2 m/s to prevent sedimentation which starts at < 0,5 m/s. The operating pressure of the membranes is limited to 3 000 kPa to prevent membrane compaction. The membranes are protected from over pressurization by the installation of bursting discs which rupture at 5 000 kPa.

The proportion of the total feed water to the bank that is withdrawn as product is termed the "pass conversion". The pass conversion on the MLT membrane bank was limited to 20 per cent to prevent precipitation of calcium sulphate scale on the membrane walls.

- (d) Back Pressure Valve. The pressure of the module bank is controlled by a manually adjusted needle valve. A short narrow diameter spool pipe is installed immediately ahead of the valve to reduce the upstream pressure of the valve and prevent cavitation.
- (e) Hydrocyclone. A 50 mm (two inch) hydrocyclone is installed on top of the reactor to concentrate the solids in the reject stream. The overflow from the hydrocyclone has a low concentration of solid particles whereas the underflow stream has a higher concentration. Smaller particles tend to enter the overflow stream and larger particles pass into the underflow stream. The efficiency of the hydrocyclone, which is affected inter alia by its dimensions and the flow rate, is characterized by the "d<sub>50</sub> cut-off", this is the size of a particle in micrometers (10<sup>-6</sup>m) which has an equal probability (i.e. 50 %) of going into either the overflow or underflow streams.

The underflow and overflow streams are diverted either to the reactor or to the purge drainage system by valves which are actuated by adjustable timers.

The purpose of the hydrocyclone is to:

- Purge, from either the underflow or overflow streams, sufficient water to give the required overall water recovery from the plant, e.g. for a 90 per cent recovery, 10 per cent of the water fed to the plant from the pretreatment section must be purged to drain.
- (ii) Control the concentration of suspended solids, i.e. calcium sulphate seeds, in the reactor and the membrane modules. This is done by preferentially purging from the hydrocyclone overflow, which has a low concentration of suspended solids, and by preferentially recirculating the underflow which has a relatively high proportion of suspended solids. The target concentration of suspended solids is 20 000 40 000 mg/l and the minimum level to avoid damage to the membranes is 10 000 mg/l. If the required water recovery were maintained by purging only from the underflow, the level of suspended solids within the system would fall below 10 000 mg/l and the level would need to be maintained by "topping-up" from an external source of calcium sulphate seed.

#### 4.3 <u>SPARRO Plant</u> (Figure 8)

- (a) Raw Water Feed Tank. Water from the pretreatment section is delivered on demand to the raw feed tank. The tank which is constructed of GRP has a buffer storage capacity of 2,6 m<sup>3</sup> which is sufficient for approximately one hours operation.
- (b) Raw Mine Water Pump. Water from the raw water feed tank is delivered to the inlet manifold of the module bank at a pressure of 3 000 to 4 000 kPa. The pump (P1A) is a Mono pump model R0 1000 which delivers 1,11 l/s (4 m<sup>3</sup>/h). Since the plant operates in the range 0,55 0,69 l/s (2 2,5 m<sup>3</sup>/h), it is necessary to depressurize and recycle some of the output back to the raw mine water tank to prevent the reactor overflowing when operating the module bank at the required pressure.
- (c) Slurry Recycle Pumps. There are two pumps fitted, P2A is supplied by National Pumps and P2B by Crown Chrome Plating. Since pump P2A has had an availability of less than 10 per cent and was never fully commissioned, only pump P2B is described at this stage. The pump is the same as that fitted on the MLT plant, i.e. a triplex positive displacement pump Model 707 fitted with three chromium plated plungers with all parts in contact with water being non-corrosive. The pump operates at a fixed speed of 295 rev/min and delivers 1,0 l/s (3,6 m<sup>3</sup>/h). The pump delivers the suspension of calcium sulphate from the reactor to the inlet manifold of the module bank.



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- (d) Module Bank. The module bank contains 88 modules arranged in four sub-banks which are arranged in series and separated by manifolds. The first sub-bank contains 30 modules arranged with 10 parallel passes each with three modules in series. The second sub-bank has 24 modules arranged in eight parallel passes each with three modules in series. The third sub-bank has 24 modules in six parallel passes each with four modules in series. The fourth sub-bank has 10 modules in five parallel passes each with two modules in series. The fourth sub-bank has 10 modules in five parallel passes each with two modules in series. The fourth sub-bank was by-passed during 1990 to limit the pass conversion and prevent damage to the modules. The combined deliveries from the raw mine water pump and the slurry recycle pump enter the inlet manifold at a pressure of 3 000 4 000 kPa. The pressure is directly affected by the setting of the reject back pressure control valve, by the delivery rate of the raw mine water pump, and indirectly by the condition (flux) of the modules. Product water from each module is fed through a flexible tube into a common tundish. The pass conversion on the SPARRO plant was usually held in the range 35 40 per cent which is higher than the level of 20 per cent on the MLT plant.
- (e) Reject Back Pressure Valve. The reject stream from the module bank is depressurized and recycled to the hydrocyclone which is installed on top of the reactor. The pressure control valve is remotely controlled from the instrument panel. The pressure in the membranes is actually controlled by the reject flow rate which is controlled by the setting of the valve. The valve is preceded by an orifice plate to reduce the upstream (inlet) pressure of the valve and to prevent cavitation.
- (f) Hydrocyclone. The hydrocyclone is a 50 mm diameter Mozely cyclone and is the same as installed on the MLT plant. The function of the hydrocyclone is the same as on the MLT plant but the method of controlling the purge rate is different. The overflow and underflow from the cyclone normally discharge to the reactor. During purging, the two streams are diverted to drain on a time cycle and diverted back to the reactor when a measured quantity of water has been purged. The system is controlled by a Siemens modular programmable controller in the instrument panel.
- (g) Reactor. The reactor has a volume of 5 m<sup>3</sup>, is made of GRP, and is fitted with an agitator. The reactor only receives the reject stream from the module bank and not the raw mine water feed as is the case on the MLT plant. This means that the pretreated feed water bypasses the reactor and is not in contact with the slurry for four hours as is the case in the MLT plant.

#### 5 PROGRAMME OF WORK

The pretreatment section and the two desalination test units, i.e the MLT and SPARRO plants, were operated from February/March 1989 to August 1990 on service water pumped from the ERPM gold mine.

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After the initial commissioning phase which was completed in June 1989, every effort was made to operate all sections continuously. Sufficient operators were seconded from ERPM to cover "round-the-clock" operation on a "seven-day" working week.

As far as possible the plants were operated and monitored under fixed and consistent conditions to check membrane performance in terms of flux and salt rejections.

Equipment and procedures on all sections were compared and evaluated and modifications were made throughout to improve reliability, process control and operating costs. Particular attention was paid to methods of keeping the membrane flux and salt rejection at their specified levels and of extending the projected lifetime of the modules.

Process conditions were logged two or three times per shift and special samples were collected weekly for analysis in the COMRO laboratories. The data and knowledge gained during the several thousands of hours of operation formed the basis for the design and costing of a plant to desalinate 5,78 l/s (500 kl/d) of scaling mine water.

#### 6 PRETREATMENT PLANT RESULTS

#### 6.1 Process Availability

An analysis of the availability (i.e. the reliability) of the pretreatment plant and the causes of breakdown is given in Appendix I. The plant was generally reliable and availabilities below 90 per cent occurred only during August and September 1989. The main causes of breakdown were electric failure of the motor and switchgear of the pretreated water delivery pump, the multiple head dosing pump and pH control. An availability of 99,6 per cent was achieved in 1990. The effect of operator absenteeism was overcome in August 1989 by the training of additional operators.

#### 6.2 Operating Conditions

Graphs of process conditions are given in Appendix II.

#### 6.2.1 Raw mine water conductivity (Graphs 1A and 1B)

The conductivity of raw mine water remained steady and was nearly always in the range 150 - 250 mS/m with an average of approximately 225 mS/m during 1989 and 1990. The apparent falls in conductivity in November 1989, between 5 000 and 6 000 hours elapsed time, and in February 1990, between 500 and 1 000 hrs elapsed time, were spurious and due to a faulty conductivity meter.

#### 6.2.2 Clarifier overflow pH (Graphs 2A and 2B)

The specification of the pH of the clarifier overflow during 1989 was 9,5 to 10. During the first 2 000 hours of elapsed time, the control was poor with pH values as high as 12 or as

ar Solis sin ⊥ low as six. The control was significantly improved after 2 000 hours with pH generally in the range 8,5 to 10,5 with occasional excursions below pH of eight. No attempt was made to control the pH of the clarifier overflow after start-up in January 1990 and the pH was effectively governed by the raw mine water. The pH in 1990 was in the range of four to nine with the higher values coinciding with irregular doseage of lime underground.

#### 6.2.3 Filter outlet pH (Graphs 3A and 3B)

The specified pH of the feed to the MLT and SPARRO plants is 5 - 6. During 1989 the pH was in the range 5 - 7 and during 1990, following changes in the process, the pH was slightly lower at 4 - 6. The pH rose to dangerously high levels of 7 - 8 after 1 000 hours in 1989 as a result of malfunctioning of the clarifier pH dosing system.

#### 6.2.4 Filter outlet turbidity (Graphs 4A and 4B)

The specification is less than 1 NTU which is approximately equivalent to  $2 \text{ mg/}\ell$  TSS. During the first 2 000 hours this objective was often not achieved. However, subsequent to the upgrading of the clarifier in May 1989, control improved markedly and turbidity was on average less than 1 NTU during the rest of 1989.

The turbidity during 1990 was adversely affected by the lower clarifier efficiency caused by the discontinuation of "two stage" pH control. The turbidity was usually below 3 NTU but numerous peaks of > 5 NTU were recorded as the filters had to remove a higher concentration of suspended solids. This may also be due to the negative effect of low pH on flocculation efficiency.

#### 6.2.5 Chlorination

Chlorination was introduced in November 1989 to prevent the growth of algae in the system and particularly of biofouling on the membranes. The level of chlorine was maintained at 1 - 3 mg/ $\ell$  (total chlorine) by dosing sodium hypochlorite solution containing 14 per cent of available chlorine into the pretreatment tank upstream of the clarifier.

#### 6.2.6 Trace metals

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Certain metals including iron, manganese, strontium and aluminium are known to damage cellulose acetate membranes in the <u>conventional</u> reverse osmosis process even when present at a concentration of about  $1 \text{ mg}/\ell(4)$ .

During 1989 the concentration of these potentially harmful metals was reduced by precipitation of their oxidized hydroxides at pH 10. The effect of "two-stage" and "single-stage" adjustments on the concentration of trace metals which could damage cellulose acetate modules is shown below in Tables 3 and 4.

Parameter*	28/06/89		06/07/89		03/08/89		31/08/89		20/09/89	
	Feed	Prod	Feed	Prod	Feed	Prod	Feed	Prod	Feed	Prod
pH pH (clarifier) Suspended Solids Iron Manganese Nickel Zinc Aluminium Strontium	4,48 - 63 0,2 5,1 5,7 1,3 3,1 4,8	5,61 9,14 3 0,1 0,2 0,8 0,2 0,0 3,4	4,37 - 68 0,6 7,8 5,3 1,8 2,2 3,5	5,47 - 3 0,3 0,3 0,1 0,2 0,4 3,4	4,57 - 82 0,3 5,2 5,1 1,9 2,4 3,2	5,64 - 4 0,3 0,4 0,3 0,3 1,8 3,1	4,53 - 37 0,3 4,7 0,5 1,4 3,8 4,6	6,28 - 0 0,3 0,2 0,5 0,2 0,8 4,2	4,93 - 24 0,3 4,0 3,4 1,1 3,5 3,9	4,11 10,09 2 0,2 0,1 0,4 0,2 0,2 3,1

# Table 3 PERFORMANCE DATA FOR PRETREATMENT PLANT WITH TWO-STAGE pH ADJUSTMENT

#### Table 4 EFFECT OF "TWO-STAGE pH ADJUSTMENT" AND "NO pH ADJUSTMENT" +

Parameter*	Two Stage p	H Adjustment	No pH Adjustment			
	Raw Water	Feed Water	Raw Water	Feed Water		
pH Suspended Solids TDS Sulphate Calcium Sodium Iron Zinc Manganese Nickel Aluminium	4,94 16 1973 1198 306 112 0,3 0,9 4,4 3,3 10	6,17 0,5 2034 1101 322 190 0,3 0,2 0,2 0,2 0,4 0,5	4,49 95 2026 1214 256 111 0,7 1,8 8,9 4,9 13.0	4,56 5 1991 1188 250 122 0,4 1,7 8,6 4,8 12 5		

+ Typical values.

\* All except pH in units of mg/l.

#### 6.3 Modifications and Plant Development (Figure 5 and 6)

6.3.1 Clarifier. The clarifier was initially incapable of operating at an output of 1,1 1/s without significant floc carry over. The clarifier was fitted with a single flocculating tube assembly in May 1989 to permit a higher upward water flow rate<sup>(16)</sup>. The flocculating efficiency was improved by the relocation of the neutralizing tank for use as a preseeding tank immediately upstream of the clarifier. The potassium permanganate and polyelectrolyte tanks were also relocated nearer to the clarifier and the dosing points moved to the clarifier feed line. These changes led to a significant improvement but the clarifier still needed careful attention to prevent carry over of the floc bed. It was intended to install a continuous turbidity meter on the clarifier overflow to provide warning of clarifier malfunction but this was not commissioned.

The flocculant was changed from ANIKEM 4816 to MAGNAFLOC 1011 to improve the consistency of the flocs as well as to improve the blow-down. A by-pass was installed to test whether the filters were capable of handling the solids load created by natural suspended solids and the precipitated heavy metals. However, it was found that the filter became overloaded within a short period and that a clarifier was essential.

#### 6.3.2 pH Control

During 1989, the pH of the feed water to the clarifier was raised to pH 10 to precipitate the iron, manganese, and aluminium hydroxides in the clarifier. The adjustment was effected by dosing caustic soda solution into the preseeding tank. The pH of the clarifier overflow was then reduced to pH 5 - 6 by dosing sulphuric acid into the filter feed tank. (Section 6.2.6).

After start-up in January 1990, the pH was no longer adjusted to pH 10 to precipitate the metal hydroxides. The caustic dosing was transferred to the filter feed tank and was readjusted to control the filter feed pH to 5 - 6. The sulphuric acid dosing system was also retained in the filter feed tank to control pH in the range of 5 - 6. After April 7 1990, no attempt was made to control the pH at all and the pH remained within acceptable limits.

#### 6.3.3 Potassium Permanganate Dosing

During 1989, potassium permanganate was dosed into the clarifier feed to oxidize iron, manganese, and aluminium to a higher valency to form an insoluble hydroxide which would precipitate in the clarifier. The dosing of potassium permanganate was discontinued in January 1990 with no negative effect on membrane life.

#### 7 MLT PLANT RESULTS

Graphs of process conditions and derived data are given in Appendix II.

#### 7.1 Process Availability

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An analysis of the availability of the MLT plant and of the causes of breakdown is given in Appendix I. If planned shutdowns are allowed for, the availabilities achieved range from 41 per cent to 92 per cent with an average of 76 per cent, while the average availability in 1990 was 82 per cent. The main contributory causes of lost time excluding the pretreatment plant, planned stoppages and operator absences were: slurry feed pump, reject control valve, reactor agitator, and power failures. The slurry feed pump was the main reason for lost time.

#### 7.2 **Operating Conditions**

#### 7.2.1 Feed water conductivity (Graphs 5A and 5B)

The conductivity of the calcium sulphate slurry feed stream to the membrane module bank was within the range 1 000 - 2 000 mS/m for the first 1 000 hours of operation. The conductivity fell to 800 mS/m between 1 000 and 2 000 hours and gradually declined to 400 mS/m by the end of 1989. This was because of the high loss of salinity from the system in the product stream as a result of low salt rejection of the modules.

The feed conductivity rose in January 1990 after new modules were installed and fluctuated in the range 500 mS/m to 900 mS/m. The feed conductivity is not directly controlled but is governed by the water recovery (i.e. purge rate) and by the condition of the modules (i.e. salt rejection).

#### 7.2.2 Feed water TSS (Graphs 6A and 6B)

The specification of feed suspended solids is 20 000 - 40 000 mg/ $\ell$  with a minimum concentration of 10 000 mg/ $\ell$ . The TSS concentration is controlled by selective purging from the hydrocyclone underflow and overflow streams. During 1989 and 1990, the concentration of calcium sulphate was within the range 10 000 to 40 000 mg/ $\ell$ . The concentration of TSS fell below 10 000 mg/ $\ell$  on several isolated occasions without causing noticeable harm to the modules; this was because the concentration of TDS in the feed had been so diluted by purging with fresh water that the process was not operating under scaling conditions at these times.

7.2.3 Feed pH (Graphs 7A and 7B)

The specification for the pH of the feed to the module bank is 5 - 6. The control during the first 1 200 hours in 1989 was poor after which the control was improved within a range of pH 4 - 7. After the "single-stage" control system was introduced in January 1990, pH control remained between pH 4 and 7 till April 1990. No control at all was applied between April and August 1990 and the pH remained steadily in the range 4 - 6.

#### 7.2.4 Product conductivity (Graphs 8A and 8B)

The product conductivity was very low (10 mS/m) at the beginning of the test because the new membranes still had a high salt rejection, but steadily deteriorated to 80 mS/m by 2 000 hours. The product conductivity rose rapidly at 5 000 hours and remained in the range 120 - 200 mS/m till the year end. The rise correlates with the rise in flux and drop in salt rejection due to membrane degradation.

The product conductivity fell sharply following the replacement of half the module bank with 10 new modules in January 1990 and remained within the range 10 - 100 mS/m. The main variations in conductivity were due to gradual build-up in feed conductivity, followed

by a sharp drop after purging. The product conductivity is directly governed by the condition of the modules as defined by their "salt-rejection". The performance of the modules during 1990 was much better than in 1989 following changes to the pretreatment system, and improved methods of membrane preservation with formalin during long plant shutdowns.

#### 7.2.5 Salt rejection (Graphs 9A and 9B)

The salt rejection at the beginning of the trials in March 1989 was very high, i.e. about 98 per cent but rapidly deteriorated over the first 2 000 hours to 87 per cent where it remained until 4 000 hours. The rejection fell sharply between 4 000 and 5 500 hours and then stabilized at 60 per cent till 7 000 hours in December 1989. The two sharp falls in rejection at 1 000 hours and 5 000 hours coincided with long plant shutdowns. It is probable that the drop in rejection during these shutdowns was due to algal fouling and inadequate preservation techniques. The introduction of chlorination in November may have prevented further loss of rejection.

Following the replacement of the 10 worst modules with 10 new modules in January 1990, the rejection rose to 90 - 95 per cent and remained fairly constant until the plant was shut down in August 1990 after a further 5 500 hours of operation. The apparent fall in rejection during the first 1 000 hours of operation in 1990 is spurious and is due to a faulty conductivity meter.

The rejection showed no sign of decline during 1990 and there is no reason to suppose that modules would fail because of hydrolysis within two years. This view is supported by Membratek, the suppliers of these modules

#### 7.2.6 Corrected flux (Graphs 10A and 10B)

The membrane flux is corrected for temperature and pressure to the standard conditions of 25 ° C and 4 000 kPa. The manufacturers design flux is 550  $l/m^2d$  at the standard condition.

The flux started at 500 -  $600 l/m^2 d$  in 1989, then increased steadily to about 750  $l/m^2 d$  and remained approximately at this level from 2 000 to 4 200 hours. The flux then rose sharply at 5 000 hours and restabilized at 900 - 1 000  $l/m^2 d$  for the remainder of 1989. The rises in flux exactly coincided with the fall in salt rejection which is a symptom of membrane hydrolysis (Table 13).

The flux remained high in 1990 when 10 new modules were installed, and then gradually declined to a constant level of 500 -  $600 \label{m2}$ .d from 2 000 to 6 000 hours when the plant was shut down in August 1990. The stability of the flux at the design value of 500 -  $600 \label{m2}$ .d indicates that the modules were not fouled, hydrolysed, or perforated.

#### 7.2.7 Product water recovery

The average product water recovery during 1989, as estimated by daily spot checks, was 93 per cent. Following the installation of water meters in March 1990, the average water recoveries were as shown in Table 5.

#### Table 5 MLT PLANT WATER RECOVERY

Month	Water Recovery %					
	Gross	Nett				
April 190	95	84				
May 1990	93	92				
June 1990	92	85				
July 1990	98	92				
August 1990	93	91				
Average	94,2	88,8				

The water recovery is defined as:

gross w.r. =  $\frac{product}{feed} \times 100 \%$ 

nett w.r. = 
$$\frac{product}{feed + flush} x 100 \%$$

The nett recovery is lower than the gross recovery because the quantity of water used to flush the modules is included with the quantity of the feed water in the denominator.

#### 7.2.8 Analysis of feed, product, and brine streams

The pretreatment system was re-evaluated in December 1989 and it was decided to discontinue the removal of iron, manganese strontium and aluminium. This decision has been supported by the improved performance of the membranes in 1990.

Typical results for the period May to September 1989 are shown in Table 6. The effect of "high" vs "low" levels of water recovery on the composition of the feed and the product streams is shown in Table 7. The feed water concentration with "low" water recovery is lower than with "high" water recovery.

#### Table 6 MLT PLANT PERFORMANCE DATA

Parameter	T	19/05/89		28/06/89		09/08/89			20/09/89			
	Feed	Prod	Brine	Feed	Prod	Brine	Feed	Prod	Brine	Feed	Prod	Brine
рH	5,54	5,73		6,23	6,55	6,27	4,82	5,51	5,01	7,32	7,09	7,47
Suspended solids (mg/3)	16 750	1	21 924	20 168	2	26 392	12 983	0	· ·	9 048	2	.
TDS (mg/4)	8 052	763	9 7 17	7 014	102	8 542	5 949	532	6 221	3 233	552	3 791
Calcium (mg/las Ca)	320	5	422	433	7	467	478	20	522	500	63	629
Sodium (mg/Las Na)	1 758	193	2 311	1 000	26	1 130	900	118	1 011	389	87	450
Sulphate (mg/las SO <sub>4</sub> )	4 752	343	6 213	3 989	42	4 937	3 150	213	3 622	1 852	222	2 140
iron (mg/las Fe)	0,3	0,2	0,6	0,2	0,1	0,2	0,4	0,3	0,4	0,2	0,1	0,2
Zinc (mg/las Zn)	0,8	0,1		1,2	0,1		1,1	0,2		0,2	0,1	0,2
Manganese (mg/Las Mn)	3,1	0,2		8,9	0,1	9,8	3,1	0,2	3,6	0,3	0,1	0,3
Nickel (mg/las Ni)	2,8	-		8,9	-	•	2,7	-	-	0,7	-	
Aluminium (mg/las Al)	4,0	0,3	.	1,0	0,0	1 .	8,0	0,2	-	0,2	0,1	
Strontium (mg/las Sr)	17,5	0,8	-	10,3	0,1	l -	8,6	0,4	-	11,4	1,1	•

### Table 7 PERFORMANCE OF MLT PLANT IN TERMS OF REJECTION OF INDIVIDUAL IONS, pH AND TDS

Parameter*	Low	Water Reco 09/02/90	overy	High Water Recovery 28/03/90			
	Feed	Product	Reject	Feed	Product	Reject	
pH TDS Ca Na SO4 CI Fe Mn	5,04 5141 722 372 2841 149 0,4 9,4	5,52 296 19 39 75 28 0,3 0,3	5,02 6240 733 450 3514 184 0,4 11,9	4,62 7734 428 767 4990 191 0,4 27,5	5,07 342 16 77 155 17 0,1 0,7	4,69 9013 450 911 5971 235 0,4 32,2	

\* All except pH in units of mg/l. Low recovery 75 - 85 % High recovery 85 - 95 %.

#### 7.3 Modifications and Plant Development (Figure 7)

#### 7.3.1 Slurry feed pump

Most of the lost time on the MLT plant was due to breakdown and maintenance of the slurry feed pump. In order to reduce lost time and maintenance costs, various types of plungers and gland packings were evaluated.

The performance of the seals and plungers used in the Crown Chrome Triplex pump is summarized in Table 8. The original CCP rope packing used with the pump was found to have a relatively short lifetime, i.e. about 300 - 400 hours, and required considerable downtime for repacking, i.e. two hours. The pump was modified to accept Chevron seals which have a lifetime of up to 500 hours and can be quickly replaced, limiting downtime to less then one hour.

• •• #
Three types of plungers were evaluated, i.e. new stainless steel (R180/plunger), chromium plated and stainless steel sleeved. The potential lifetime of each of these types is approximately 1 000 hours. The cost of a rechromed plunger is R70 each, and the cost of resleeving is R60 each (i.e. labour R50 and material R10). The full life of the plungers was not utilized as they were changed prematurely whenever the packings were changed, to avoid damage to the new packings and to minimize the maintenance time and consequent loss of production. The material cost of maintaining the plungers and packings is 22,8c/kl. The use of very hard plunger surfaces such as ceramics has not been investigated to date but may be attractive if the packing wear can also be overcome.

The rate of wear of plungers fitted with stainless steel pipe sleeves was compared to that of chromium plated plungers and it was found that chromium wears at almost half the rate of a stainless steel surface.

Date	Pump Hours	Plunger Hours	Maintenance
13.10.89	9262	-	Extra packing to plunger 1,3.
16.10.89	9290	1732	Replace plungers 1,3; New CCP (rope) packing.
10.11.89	9797	1088	Replace plunger 2; New chevron seal.
20.11.89	9992	702	Replace plungers 1, 3; Repack with CCP packing.
27.11.89	10124	327	Replace plunger 2; New chevron seal.
09.12.89	10341	-	Extra CCP in 1, 3; New chevron seal in 2.
16.01.90	10544	-	Glands 1, 3 to take chevron seals; Resleeved plungers installed in 1, 2,3.
23.01.90	10635	-	Replace chevron seals 1, 2.
12.02.90	11062	518	Resleeved plunger installed in 1, 2, 3,; New chevron seals.
18.03.90	11849	787	Resleeved plungers installed in 1, 2, 3.; New chevron sleeves
04.04.90	12195	346	Rechromed plungers installed in 1, 2, 3.; New chevron seals
30.04.90	12627	432	Resleeved plungers installed in 1, 2, 3.; New chevron seals
13.06.90	13214	587	Pump overhauled, new plungers fitted
20.07.90	13746	532	Changed plungers

#### Table 8 MLT PLANT SLURRY FEED PUMP PLUNGER LIFE

#### 7.3.2 Reject pressure control valve

The reject pressure control valve was changed from automatic to manual operation following problems with the control loop in September 1989. The modified system was more stable and easy to operate.

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#### 7.3.3 Module bank

The module bank was fitted with 20 new modules in March 1989 at the beginning of the trials. The salt rejection of each module was measured on 25 occasions during the following 18 months of operation and is recorded in Appendix III. The date of installation and removal is also recorded. A summary of the frequency of membrane replacement is given in Table 9.

The performance of the membranes during 1989 was unacceptable as indicated by measurements of salt rejection, product conductivity and membrane flux. Samples of membranes were removed from the stack after 1 000 and 2 000 hours of operation and sent to the University of Stellenbosch Institute for Polymer Science (USIPS) for inspection. The results are shown in Appendix VII and it was concluded that:

- . the membranes appeared to be physically eroded,
- . surface deposits containing mainly aluminium silicate were observed,
- . the membranes appeared to be hydrolysed.

Half of the modules in the bank were replaced in January 1990 as their salt rejections had declined to 50 per cent and the corrected flux had risen. The reasons for the module degradation include hydrolysis and possibly biological fouling.

The situation was much improved during 1990 and the performance of the module bank was acceptable following changes in the pretreatment system.

		MEMBRANE REPLACEMENTS																	
POSITION	'89 Mar.	'89 Apr.	'89 May	'89 June	'89 July	'89 Aug.	'89 Sept.	'89 Oct.	'89 Nov.	'89 Dec.	'90 Jan.	'90 Feb.	'90 Mar.	'90 Apr.	'90 May	'90 Jun.	'90 July	'90 Aug.	Total
1	1			2							1								4
2	1										1								2
3	1							1											2
• 4	1							1			1					1			3
5	1		1	1			1												3
8	1							·			1	1			1				4
7	1										1								2
8	1									1									2
9	1										1								2
10	1				1		·							1					3
11	1								1		1								3
12	1								1			1					1		4
13	1								1				1						3
14	1								1			1							3
15	1				1		1		1					2					6
16	1										1	1	_						3
17	1										1	1	1	1					5
18	2							1											3
19	1	1						1			1								4
20	1									1	1								3
TOTAL	21	1	0	3	2	0	2	3	5	2	10	5	3	4	1	1	1	0	64

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#### Table 9 MLT PLANT MEMBRANE REPLACEMENT

During the 18 months of operation of the 20 module stack, a total of 64 modules were used. The reasons for replacement are given below:

Mechanical failure (23). This includes blocking and failure by bursting.

Low salt rejection (15). Modules were replaced in November 1989 (5) and in January 1990 (10). This was as a result of the poor pH control and inadequate preservation procedures in 1989.

Samples for analysis (6). Samples were sent to the University of Stellenbosch Institute for Polymer Science (USIPS) for analysis.

Initial installation	20
Mechanical failure	23
Low salt rejection	15
Samples for USIPS	<u>6</u>
Total	64
Modules in use 31/08/90	<u>20</u>
Modules used in trials	44

Average lifetime of a module before failure by bursting or blocking was 15,6 months  $(18 \times 20/23)$ . The longest operating module in use on 31 August 1990 had been in use for 5 421 plant hours. Production problems which led to mechanical failure of modules were identified and rectified by Membratek.

#### 7.3.4 Hydrocyclone

The hydrocyclone installed on the MLT plant was 50 mm in diameter. The control of the diversion of the overflow and underflow to drain was by automatic timer and the system remained unchanged.



# Figure 9 MLT PLANT AND SPARRO PLANT CaSO<sub>4</sub> SEED SIZE IN FEED TO CYCLONE

The size distributions for the particles recirculated in the plant were analysed by COMRO and  $CSIR^{(15)}$ . The average size of the seed in the feed to the cyclone as measured by COMRO between September 1989 and March 1990 is given in Figure 9.

Detailed results obtained by COMRO are given in Appendix IV.

A microscope photograph of conditioned seed from the MLT plant is given in Appendix VI.

## 8 SPARRO PLANT RESULTS

#### 8.1 Process Availability

An analysis of the availability of the SPARRO plant and the causes of breakdown are given in Appendix I.

If planned shutdowns are discounted the availabilities range from 42 to 86 per cent with an average of 67 per cent. The availability in 1990 was 70 per cent. The main reasons for lost time excluding the pretreatment plant, planned stoppages and operator absence were: the slurry recycle pump, module replacement, module cleaning, module renovation, and instrumentation.

#### 8.2 **Operating Conditions**

#### 8.2.1 Feed water conductivity (Graphs 11A and 11B)

The feed water conductivity remained within the range of 200 and 400 mS/m throughout 1989, except for a brief initial period at 800 mS/m. This low range of values is attributed to the low salt rejection of the modules. The conductivity rose in 1990 and remained in the range 600 - 800 mS/m after the new modules were installed and the size of the module bank was reduced.

#### 8.2.2 Total suspended solids (Graphs 12A and 12B)

The level of suspended solids during 1989 was between 2 000 and 25 000 mg/ $\ell$ . This parameter was difficult to maintain because the low salt rejection and feed conductivity inhibited the growth of calcium sulphate seed particles. It was frequently necessary to boost the seed concentration with surplus seed from the MLT plant and by adding commercial gypsum. The addition of commercial gypsum is not recommended because it blocks the inlet manifold of the slurry delivery pumps.

Following the replacement of the membranes in January 1990 and the adoption of procedures to maintain the efficiency of the modules, the TSS generally remained within the range 10 000 to 50 000 mg/ $\ell$  with an average of approximately 25 000 mg/ $\ell$ . However, on 8 July the seed concentration accidentally fell below 10 000 mg/ $\ell$  and this led to an immediate rise in the flux and a corresponding fall in the salt rejection. This process accident verifies the previously assumed lower concentration limit of 10 000 mg/ $\ell$  for normal operation with saturated feed water. This problem may also have occurred in 1989 but was not detected because of the poor condition of the module bank.

#### 8.2.3 pH of feed (Graphs 13A and 13B)

The pH of the feed remained in the range four to seven with an average of 5,5 during 1989. The pH in 1990 was usually lower and most frequently in the range of 4 - 5. Short peaks of as high as 7 - 8 occurred which corresponded to the use of high pH flush water.

8.2.4 Product water conductivity (Graphs 14A and 14B)

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The conductivity started at 100 - 140 mS/m and then rose to 160 - 190 after 3 000 hours when the modules were clearly degraded and exhibiting a low salt rejection. Module degradation is attributed to biofouling, poor pH control, high pass conversion and inadequate preservation during shutdowns (Table 13). Following the installation of 88 new modules in January 1990, the conductivity was initially 80 - 100 mS/m. The conductivity soon started to rise and reached 140 mS/m within 500 hours before the bottom section of the module bank was isolated to reduce the pass conversion. The

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conductivity then fell and remained in the range 30 - 100 mS/m during 1990 with an average of approximately 70 mS/m. This level was governed by the salinity of the feed stream and the purge rate.

#### 8.2.5 Salt rejection (Graphs 15A and 15B)

The membranes installed at start up in 1989 were not new. The initial rejection was 80 - 90 per cent but this steadily declined and had reached 50 per cent by the end of the year after 6 500 hours elapsed time due to module degradation (Table 13). The rejection was increased to 80 - 90 per cent after new modules were installed in January 1990, but this fell rapidly to 55 per cent after 500 hours. As a result of the isolation of the fourth section of the module bank, the salt rejection recovered and remained close to 90 per cent until shutdown in August after a further 4 000 hours had elapsed. The rejection dipped by approximately five per cent after 5 000 hours as a result of a brief period of operation with a TSS of less than 10 000 mg/ $\ell$ . The modules were renovated and the rejection rose to 90 per cent and remained at that value till the end of the trials at 5 000 hours. The renovation procedure is discussed in Appendix VII.

#### 8.2.6 Corrected Flux (Graphs 16A and 16B)

The flux increased to 1 000  $\ell/m^2$ .d during the first 2 000 hours and then to 1 300  $\ell/m^2$ .d after 4 000 hours, and stayed at this value till the end of 1989 (6 000 hours).

The high flux, which is approximately double the design value of 500 - 600  $\ell/m^2$ .d, shows that the modules had been degraded and coincides with the low salt rejection values during this period (Table 13). Following the replacement of the module bank in January 1990, the flux rose rapidly and erratically from 800 to 1 000  $\ell/m^2$ .d after 500 hours. When the fourth bottom bank of the modules was isolated, the flux fell immediately to 550  $\ell/m^2$ .d, indicating that the damaged modules were all in the bottom bank. The flux declined from 500  $\ell/m^2$ .d in April to 300  $\ell/m^2$ .d in June 1990 because of fouling. After the modules were cleaned with Biotex (1 %) and citric acid (2 %) the flux rose to 400  $\ell/m^2$ .d on 14 June. The flux declined again to 300  $\ell/m^2$ .d on 7 July when the seed concentration fell below 10 000 mg/ $\ell$ . The flux then rose to 380  $\ell/m^2$ .d on 12 July and 420  $\ell/m^2$ .d which fell to 350  $\ell/m^2$ .d on 16 August and remained steady at this level till shutdown on 31 August.

The SPARRO plant appears more susceptible to membrane fouling than the MLT plant, probably as a result of the foulants not being removed from the feed by contact with calcium sulphate seed in the reactor.

#### 8.2.7 Product water recovery

The product water recovery was estimated by regular comparison of the purge rate with the plant feed rate during 1989 and up to May 1990 as no water meters were installed. Monitoring of the water recovery started in June 1990 and was delayed by difficulties in installing the water meter in the product line.

The results of the spot checks and the more accurate metered measurements show that the nett water recovery on the SPARRO plant was between 93 and 97 per cent.

#### 8.2.8 Analysis of feed, product and brine streams

Typical analyses of the composition of the feed, product and brine streams are shown in Table 10. As stated in Section 7.2.8, the concentration of iron, manganese, strontium and aluminium was not considered to be so important in 1990 following the discontinuation of the "two-stage" pH adjustment system.

The effect of high feed salinity at high water recovery vs low feed salinity at low feed salinity is shown in Table 11.

Parameter		21/08/89		11/07/89			23/08/89			22/09/89		
	Feed	Prod	Brine	Feed	Prod	Brine	Feed	Prod	Brine	Feed	Prod	Brine
рH	6,39	6,46	6,77	6,48	6,25	6,79	5,93	5,85	5,83	7,32	7,31	7,35
Suspended solids (mg/4	12 040	0	21 438	9 400	0	16 290	9 767	0	16 638	13 496	1	32 748
TDS (mg/4)	4 355	1 253	6 107	4 352	1 398	5 738	3 605	1 800	4 502	3 3 1 4	1 804	3 851
Calcium (mg/Las Ca)	458	100	440	461	72	467	544	206	556	489	200	544
Sodium (mg/タas Na)	542	158	829	683	311	994	433	256	578	406	227	556
Sulphate (mg/las SO4)	2 354	643	3 4 3 6	2 373	788	3 220	1 975	869	2 500	1 864	1 017	2 246
Iron (mg/las Fe)	0,2	0,1	0,2	0,3	0,3	0,4	0,3	0,2	0,4	0,3	0,3	0,3
Zinc (mg/las Zn)	0,5	0,3	0,6	0,4	0,3	0,5	0,3	0,2	0,4	0,6	0,2	0,3
Manganese (mg/las Mn)	0,8	0,3	1,1	0,7	0,2	1,1	0,5	0,2	0,9	0,2	0,1	0,2
Nickel (mg/Las Ni)	0,5	-	0,7	0,4	•	0,8	0,5	-	1,0	0,5	•	0,5
Aluminium (mg/las Al)	1,4	0,4	1,1	2,7	0,0	0,0	1,5	0,5	2,7	1,5	0,0	0,0
Strontium (mg/Las Sr)	7,2	1,5	10,2	8,4	2,7	13,0	6,1	2,5	7,9	7,0	3,3	9,8

#### Table 10 SPARRO PLANT PERFORMANCE DATA

Parameter*	Low	Water Reco 07/03/90	overy	High Water Recovery 28/03/90			
	Feed	Product	Reject	Feed	Product	Reject	
pH	4,44	4,96	4,44	5,35	5,45	5,23	
TDS	5370	299	5660	11229	599	17255	
Ca	511	17	656	433	19	422	
Na	389	57	378	911	113	1411	
SO₄	3500	131	3568	7853	278	12001	
Cl	132	27	175	101	46	357	
Fe	0,3	0,2	0,2	0,3	0,2	0,4	
Mn	26	0,9	23	47	1,7	66	

## Table 11 PERFORMANCE OF SPARRO PLANT IN TERMS OF REJECTION OF INDIVIDUAL IONS, pH AND TDS

\* All units in mg/l except pH. Low water recovery 90 - 95% High water recovery 95 - 99%

## 8.3 Modifications and Plant Developments (Figure 8)

#### 8.3.1 Mine water pumps P1

The two multi-stage centrifugal pumps (ex ISCOR SRO plant) which were initially fitted were mechanically unreliable and difficulties were experienced in obtaining replacements from the USA for the badly eroded impellers. The two pumps were replaced with a locally supplied Mono pump (Model RO-100) which is specially designed for reverse osmosis applications and constructed of a corrosion resisting steel. The Mono pump (P1A) proved very reliable and required only 74 hours of maintenance between June 1989 and August 1990, which gives an availability of over 99 per cent. The cause of the breakdown was due to the failure of the process instrument air supply which caused a restriction in the supply of water to the suction of the Mono pump.

#### 8.3.2 Slurry recycle pumps

The slurry recycle pumps were the cause of most of the lost time. The National pump P2A was unreliable throughout, while the Crown Chrome pump (P2B) exhibited prolonged uninterrupted operation during the final six months of the trials in 1990. The initial problems experienced with the Crown pump P2B were inexplicable because an identical pump was operating well on the MLT plant in the same duty. The problems experienced are listed below.

Air locking. The problem was manifested by a gradual loss of output after several hours of normal operation. The immediate problem was relieved by removing and cleaning the suction sieves, removing the valve covers and flushing out the air bubbles with water.

- . Slow pick-up to full delivery rate. The pump took up to one hour to reach full output and the National pump P2A took longer. The problem was probably due to the presence of air bubbles in the cylinders.
- . Pressure pulsations. This led to bursting disc failures.
- . Gland packing maintenance. The time required for replacement of the rope packings was two hours.
- . Blockage of inlet manifold. The inlet manifolds of the pumps were frequently blocked with settled calcium sulphate seed.

The following remedial actions were taken:

- a. National Pump P2A. The pump operated well in only one month October 1989 when the Crown pump glands were being modified to take "chevron" seals. The pump was serviced in November 1989 by the suppliers and the glands modified to take PTFE (Teflon) packings. The pump was used again in February 1990 and could not achieve the required output. The valve seats were then reground by COMRO and the pump put back into use. The pump operated for only 44 hours before it was taken out of service again. The pump was overhauled again by the suppliers, but was only available for use in September 1990 after the SPARRO plant trials had been completed and therefore could not be re-evaluated in situ.
- b. Crown Pump P2B.
  - (i) The output of the pump was uprated by 21 per cent from  $0.85 \label{eq:loss}$  to  $1.01 \label{eq:loss}$  by increasing the speed from 243 to 295 rev/min. Although this was done primarily to increase the feed rate to the membrane bank it may have been beneficial in the prevention of manifold blockages.
  - (ii) The "rope" packings used in the gland seals were replaced by rubber "chevron" seals in October 1989. This reduced the time required for gland maintenance to less than one hour in 500 hours.
  - (iii) The inlet manifold was drained each shift to prevent the accumulation of calcium sulphate seed which could restrict the feed of slurry to the pump.
- c. Instrument Air Pressure. The diaphragm valve between the reactor and the Crown pump P2B was held open by the pressure of the instrument air supplied by ERPM gold mine. The pressure of the air must be 500 kPa to fully open the valve. It was observed that the air pressure frequently fell below 300 kPa, especially at night. The partial closure of the valve increases the hydraulic resistance between the pump suction and the reactor and reduces the NPSH (net positive suction head) below the

required 1 m (w.g). This could cause the ingress of air into the pump and the observed loss of output. The problem was overcome by replacing the unreliable mine air supply with bottled nitrogen gas.

- Suction Valve. The valve at the suction of the Crown pump was found to be defective and to have an air leak in the diaphragm which restricted the degree of opening. This was replaced.
- e. Addition of Gypsum Seed. The addition of gypsum seed to the reactor to boost the level of suspended solids was frequently carried out in 1989 because of the poor seed growth conditions, and was occasionally carried out in 1990 following excessive purging. It was observed that this was usually followed within several hours by a loss in output from the pump. The reason is that the large and irregular crystalline particles settle out quickly and form a stiff paste in the inlet sieves and inlet manifold. The use of commercial gypsum has been discontinued. A microscope photograph of coarse gypsum is shown in Appendix VI.
- f. Reactor Level. It was observed that if the level in the reactor fell below 1 m from the top, then the output of the pump fell. The reactor level was therefore maintained as high as possible without overflowing.
- g. Alternative Plungers/Packings. The lifetime of the plungers and packings in the Crown pump are shown in Table 12. The results were consistent with those given in Table 5 for the MLT plant. It appears that chromium plated plungers are the most cost-effective.

Date	Pump Hours	Plunger Hours	Maintenance
09-11-89	988	-	Modified to use chevron seals, new plungers 1, 2, 3
09-12-89	1498		Replace chevron seals 1, 2, 3
16-02-90	1810	822	Replace chevron seals 1, 2, 3 replace plungers 1, 2, 3
02-03-90	1902	92	Fit sleeved plungers and packing 1, 2, 3
07-03-90	1996	-	Replace faulty plungers (not round) and chevron seals
31-03-90	2350	448	Replace plungers & packing 1, 2, 3 with hard chromed plungers
30-04-90	2779	429	Replace plungers & chevron seals (plungers - resleeved)
14-05-90	3033	254	Replaced plungers & chevron seals
18-05-90	3115	82	Replaced plungers 2, 3 & chevron seals 2, 3
25-05-90	3272	1 2 (239) (157)	Replaced plunger 1, 2 chevron seals 1, 2
05-06-90	3382	267	Replace plunger 3 chevron seal 3
20-06-90	3653	1 2 3 (381) (381) (271)	Replace plungers 1, 2, 3 chevron seals 1, 2, 3
02-07-90	3921	268	Replace plunger 3 chevron seal 3
09-07-90	4064	411	Replace plunger 2 chevron seal 2, 3
10-08-90	4231	578	Replace plunger 1 chevron seal 1
17-08-90	4356	-	Replace chevron seal 3
30-08-90	4628	•	Plant shutdown

#### Table 12 SPARRO PLANT SLURRY FEED PUMP PLUNGER LIFE

#### 8.3.3 Module bank

The initial 88 modules installed on the SPARRO plant in February 1989 were not all new as some of them had been used previously by ISCOR. As described in Section 8.2.4 and 8.2.5, the membranes degraded within the first 2 000 hours of elapsed time. It was decided, however, to leave the modules in to gain operating experience with the various novel design features of the SPARRO plant. It was also anticipated that the causes of membrane degradation would be identified and rectified. Following the changes to the pretreatment section, the introduction of chlorination in November 1989 and the adoption of improved module preservation procedures, a completely new set of 88 membranes was installed in January 1990. The modules were operating in the tapered four bank mode when the plant was restarted on 27 January as shown below.

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Sub-bank	Position	Modu	Total	
		Parallel	Series	
1	Top inlet	10	3	30
2	Upper middle	8	3	24
3	Lower middle	6	4	24
4	Bottom outlet	5	2	10
Total				88

It became apparent within the first 1 000 hours that the modules in the fourth sub-bank (Nos 79 to 88) were showing a rapid loss in salt rejection and an increase in flux. This caused the product water quality to deteriorate. Microscopic inspection of the membranes showed perforation and crystal growth in the membranes. Microscopic photographs of the affected modules are given in Appendix V. The damage was attributed to the development of an excessively high degree of calcium sulphate supersaturation at the end of the module stack in sub-bank 4. The level of calcium sulphate supersaturation is affected inter-alia by the pass conversion of the module bank. The value of pass conversion adopted in the design of the SPARRO plant was 50 per cent, with a minimum value of 40 per cent based on previous pilot plant work. The actual value of the pass conversion during initial operation may have been as high as 60 per cent because of the high flux. The level recommended by Resources Conservation Company International (RCCI) based on work carried out for the Electric Power Research Institute (EPRI) was 30 - 40 per cent(10).

The pass conversion was reduced by removing the damaged bottom sub-bank of 10 modules and by increasing the output of the slurry delivery pump P2B from 0,85  $\ell$ /s to 1,0  $\ell$ /s. The reduction in module area of 11 per cent caused the product rate to fall to 0,69  $\ell$ /s and the output of the pump rose from 0,85  $\ell$ /s to 1,0  $\ell$ /s. The combined effects of the changes caused a reduction in pass conversion to 40 per cent. The pass conversion gradually declined from February as the modules became fouled, and the product rate fell, and by April the pass conversion had reached 30 per cent.

The tapered stack performed reasonably well, but it was apparent that there were some distribution problems at the end of the inlet manifold as modules 10, 20, 30 became blocked twice in the first 1 500 hours of 1990.

The overall size of the module bank was reduced from 88 to 78 by isolating the 10 modules in the bottom sub-bank in February 1990. The isolation of modules 10, 20 and 30 in March 1990 further reduced the size to 75. As a result of a shortage of membranes in August, six more membrane positions were isolated which reduced the size to 69.

The main problem with the SPARRO module bank in 1990 was the decline in the corrected flux from 600 to  $300 \ l/m^2$ .d in June. This is in contrast to the MLT plant where the flux stabilized at the design value of 500 - 600  $\ l/m^2$ .d. The modules were cleaned in June with Biotex and citric acid solutions to remove organic and inorganic foulants.

The procedures for the cleaning of modules which were provided by Membratek are included in a separate report on the design of a 500 k<sup>l</sup>/day SPARRO plant. It is estimated that cleaning would be required every 500 hours to maintain an acceptable flux. This is probably a result of fouling of the membranes by colloidal material which has passed through the filter and which has not been absorbed by the seed particles as is thought to be the case on the MLT plant. The provision of an adequate contact period between pretreated water and the agitated slurry is an important design feature which is discussed in Sections 9 and 10.

As a result of the minor damage suffered by the module bank on 7 July 1990 when the concentration of seed fell below 10 000 mg/l, it was decided to evaluate the membrane renovation procedures which had been developed by the University of Stellenbosch Institute for Polymer Research<sup>(11)</sup>. The principle of the restoration procedure is to lay a thin film of new polymer above the damaged membrane. The film is formed by the separate addition of polyvinyl methyl ether (PVME) and tannic acid in a series of controlled pH steps as described in detail in the separate design report.

A summary of common causes of membrane damage is shown below in Table 13.

	Symptoms	Cause	Restoration Procedures
Case I	<ol> <li>Lower product water flow rate</li> <li>Higher salt rejection</li> </ol>	Membrane compaction accelerated by operating pressure greater than 3 450 kPa.	None. Requires element replacement when product water flow rate reaches an unacceptable level.
Case II	<ol> <li>Higher product water flow rate</li> <li>Lower salt rejection</li> </ol>	Membrane hydrolysis 1. pH outside operating limits 2. Bacteria degradation 3. Temperature outside operating limits.	Injection of PVME and tannin or element replacement.
Case III	1. Lower product water flow rate 2. Lower salt rejection	Membrane fouling.	Element cleaning.
Case IV	<ol> <li>Lower product water flow rate</li> <li>High △P</li> <li>High operating pressure</li> </ol>	Membrane fouling.	Element cleaning.

#### Summary of common causes of membrane damage

The inlet pressure to the modules on the SPARRO plant was usually in the range 3 500 - 4 500 kPa, which may have contributed to module compaction.

#### 8.3.4 Hydrocylone

A set of six 10 mm diameter hydrocyclones was initially installed. It was found that the walls of the cyclones were quickly worn by abrasive calcium sulphate seed. The underflow from the cyclones was sticky and dense which caused blockages. The set of

six was replaced by a single 25 mm hydrocyclone during commissioning. An attempt was made to predict the particle size distribution of the underflow and overflow streams using a computer model developed by COMRO and based on empirical cyclone modelling technology<sup>(12)</sup>, but the results showed a marked variance from prediction.

The 25 mm hydrocyclone was replaced in March 1990 with a 50 mm hydrocyclone which was identical to that installed on the MLT plant. The pump blocking problem was overcome in March 1990 but it is unlikely that this was because of the new cyclone. As a result of the installation of water meters and the more accurate determination of the water recovery rate, the operation of the hydrocyclone at a water recovery of below 96 per cent was found to be impossible because of restrictions in the pipework and valves of the purge discharge system.

#### 8.3.5 Reactor

The level control of the SPARRO plant reactor was troublesome throughout the trials. If the level rises then the reactor may overflow and the slurry may even become diluted below the lower concentration limit of 10 000 mg/ $\ell$ . If the level falls, then the available suction head to the slurry recycle pumps falls below the 1 m(wg) required and the output falls, and this may also cause air bubbles to be drawn into the cylinders.

The level in the reactor is not automatically controlled as in the MLT plant. It is inherently unstable and requires frequent operator attention to adjust the process variables which maintain a constant level. The operator had to control the level by adjustment of the raw mine water feed rate from pump P1A, and the reactor pressure by adjustment of the reject control valve. If the reactor level fell, then the reactor pressure was in turn affected by the lower recycle slurry rate. The level of the reactor on the MLT plant is automatically controlled at one level -"full"- by a float valve which admits water on demand.

The main concern in the design of the reactor was that the residence time and the agitation should be sufficient for the supersaturated slurry to reach equilibrium before it was recycled to the membranes. The required time to dissipate supersaturation was estimated to be several minutes by plotting the decline of slurry conductivity in the reject stream from the module bank. This is less than the required residence time of one hour predicted from fundamental research into the kinetics of calcium sulphate precipitation carried out by the CSIR in 1988<sup>(14)</sup>.

Measurements of seed concentration profiles in the reactor taken during December 1989 showed some maldistribution and this was rectified by installing an additional impeller in the middle of the agitator shaft.

#### 8.3.6 Reject control valve

The original method of controlling the slurry flow rate through the membrane stack was indirect by adjusting the reject flow rate to control the inlet pressure. This proved

impractical and led to difficulties in controlling the level of the reactor. The reject flow control valve was disconnected from the reactor pressure loop and was then controlled directly to give the required flow rate. The reject flow rate, the recycle slurry feed rate, the raw mine water feed rate, the condition of the membranes, and the temperature of the water interact to control the product water rate, the membrane pressure and the reactor level.

#### 9 PROCESS EVALUATION AND DISCUSSION

The evaluation of the process is focused on the primary objectives listed in Section 2. Both economic and non-economic criteria are taken into account, e.g.

- a. economic factors:
  - membrane lifetime
  - electric power consumption
  - maintenance costs.
- b. non economic factors:
  - process availability/reliability
  - control
  - flexibility.

Particular attention is paid to the comparison of the orthodox design of the MLT plant with the novel features included in the SPARRO plant. These include the twin pump delivery system, the tapered module bank and the hydrocyclone system.

#### 9.1 Pretreatment Plant

It is necessary to pretreat the water to protect the membranes in the MLT and SPARRO plants by removing materials and conditions potentially harmful to the RO process such as:

- . remove materials to prevent scaling and fouling
- . adjust pH
- . reduce temperature
- . remove turbidity/suspended solids
- . disinfect to prevent biological growth.

#### 9.1.1 Removal of iron and manganese

During the previous work<sup>(7)</sup> by COMRO, no attempt was made to remove iron or to prevent it fouling the modules in the form of iron hydroxide. It was later suggested that the module life could be extended by removal of iron and other possible contaminants such as manganese, aluminium and strontium. Although it is claimed that the slurry

reverse osmosis process does not need the removal of iron and manganese, it was decided to remove them as this had proved beneficial in tests carried out with conventional RO on non-scaling mine water<sup>(4)</sup>.

When the module salt rejection fell and the flux climbed during 1989 it was thought that this may possibly be due to the inadequate removal of iron, manganese or other unspecified trace metals. The removal of iron and manganese hydroxides was achieved by raising the raw mine water pH to 10 by the addition of caustic soda solution, together with addition of potassium permanganate to oxidise the metals.

It is evident, in the light of the acceptable performance of the modules during 1990, that the precipitation of hydroxides is not necessary. As well as being unnecessary, the removal of hydroxides at pH 10 in the clarifier is a potential hazard to the membranes, because the contents of the reactor could run through the membranes in the event of a failure of the second downstream stage of the pH adjustment and a clarifier malfunction. The cost of the two-stage adjustment process, i.e. from pH 5 to pH 10 and back to pH 5, in terms of caustic soda and sulphuric acid solution, is  $R0,16/k^2$  which is an unnecessary expense.

#### 9.1.2 Final pH

The target pH quoted by the suppliers of the membranes, Membratek, is pH 5 - 6. Examination of the cellulose acetate hydrolysis curve (Figure 3) shows that a range of pH 4 - 6 is also acceptable. The reason for quoting a range of 5 - 6 is to minimize the cost of reducing pH in alkaline water and does not imply that water with a pH 4 will damage the membranes.

The pH control system proved troublesome throughout the trials and it was dispensed with in April 1990. The problem was mainly due to the sensitivity and fragility of the probes rather than to the design of the system. It is therefore essential that the pH of the pretreated water be checked twice per shift by a laboratory pH meter which should be standardized regularly.

#### 9.1.3 Temperature

The upper limit of temperature is 30 ° C and this did not cause any problems during the trials. The temperature can be reduced by the installation of a cooling tower in the pretreated water holding tank. The effect of heat addition through the dissipation of power in the high pressure slurry delivery pumps must be taken into account.

#### 9.1.4 Removal of suspended solids

It has been claimed that it is not necessary to remove suspended solids at the pretreatment stage, because 20 000 mg/l of calcium sulphate is present in the feed to the membranes. However, it must be appreciated that the nature of the quartzitic suspended

solids in raw mine water or other possible feed may be particularly harmful. It is reported by  $RCCI^{(10)}$  that the presence of fly-ash is very damaging and that its removal was necessary to preserve membrane efficiency. It is also possible that abrasive solids could damage the high pressure pumps.

#### 9.1.5 Disinfection

Reverse osmosis modules provide a large surface area for the attachment and growth of bacterial slimes and moulds. These organisms may cause membrane fouling or even module plugging. There is also some evidence that occasionally the enzyme systems of some organisms will attack the cellulose acetate membrane. The continuous application of chlorine to produce a 1 to 2 mg/ $\ell$  chlorine residual will help inhibit or retard the growth of most of the organisms encountered. However, caution must be exercised since continuous exposure of the membrane to higher chlorine residuals will impair membrane efficiency. Shock concentrations of up to 10 mg/ $\ell$  of chlorine may be applied from time to time.

The absence of chlorination between February and November 1989 was almost certainly a major reason for the poor performance of the modules in that period. Chlorination can be achieved by dosing sodium hypochlorite solution or by injecting chlorine gas. Chlorine gas is the more economic choice for large installations.

#### 9.2 Membrane Life

The performance of the tubular cellulose acetate membranes is the key factor in the technical and economic viability of the process. The performance and projected lifetime of the modules are measured in terms of the salt rejections (based on the feed to the modules <u>not</u> to the plant) and of the corrected flux. Membranes require replacement after either mechanical failure, or when the salt rejection falls below a set level, e.g. 80 per cent, or the corrected flux falls below a set level e.g.  $400 \ l/m^2$ .d. The performance of the MLT module bank during 1990 indicates that a lifetime of at least two years (17 520 hours) is realistic provided that they are operated steadily and not mechanically damaged by blockage or bursting. Modules are guaranteed against rapid mechanical failure and will be replaced by the manufacturers.

The performance of the membranes in the SPARRO plant was not as good as in the MLT plant, mainly as a result of the fouling which occurred. The fouling is probably a result of the presence of fine solid particles and other foulants which may have passed through the pretreatment plant and which have not been removed by prolonged contact with calcium sulphate seed particles in the reactor. This is a major disadvantage of the SPARRO plant relative to the MLT plant. Nevertheless, the modules in the SPARRO plant configuration probably could achieve a lifetime of two years subject to a regular monthly cleaning with citric acid and Biotex solutions. Provisions for the periodic cleaning of the reverse

osmosis elements are included in the system design. This makes it possible to clean impurities off the membrane surface and restore normal flow rates in situ without removing the membranes from the stack.

The operating pressure has a bearing on the membrane life (Table 13). If the pressure exceeds 3 000 kPa then irreversible membrane compaction will occur. This probably contributed to the relatively inferior performance of the SPARRO plant in 1990 where the inlet pressure was usually in the range 3 500 - 4 500 kPa as the significance of the pressure was not appreciated.

Mechanical failure of the modules by blocking or by over-pressurization will probably not reduce the average module lifetime below two years on a commercial unit if the module bank is properly designed to prevent sedimentation and bypassing. It will be necessary to seek specialist advice on the design of the manifolds<sup>(13)</sup>.

#### 9.3 Tapered Module Stack

There are a number of aspects that need to be considered when considering the module configuration:

- . tapered (SPARRO) versus straight through (MLT)
- . manifolding
- . minimum velocity
- . number of modules

. .

. pass conversion.

The major disadvantages of the straight through (rectangular) system is that there is a reduction in linear velocity through the tube bundle, and thus a high velocity is required at the inlet to achieve a required velocity in the last modules. The claimed advantages of a tapered system were:

- the linear velocity is maintained through the tube bundle which allows for a lower inlet velocity and the achievement of a high pass conversion.
- in the last part of the system where the greatest concentration is taking place there are fewer modules exposed to the higher feed salinity and therefore the overall product water quality should be better.

the ability to increase the velocity of the brine in the last section of the taper reduces the risk of concentration polarization influencing the module performance. A more gradual pressure drop gives better overall fluxes. With the setting of the maximum pass conversion at 30 per cent, the main advantage of the tapered module configuration is reduced. This is illustrated in the design prepared in separate design report where the inlet velocity of 2,3 m/s declines to only 1,7 m/s at the outlet in a stack containing seven modules in series and 10 modules in parallel.

Large module banks present piping and manifolding problems when dealing with a slurry. For this reason it was decided to design the pilot plant manifold to be similar to that of an anticipated full scale unit in order to assess its effectiveness. The module manifolds did not allow for smooth changes in velocities and were complicated by the need for intermediate manifolds and valves to give the required tapering. The inlet manifold design was not optimal<sup>(13)</sup> and probably contributed to the repeated blockage of modules 10, 20, and 30. The MLT stack which had five modules in series without intermediate manifolds and valves to give the need for intermediate manifolds and valves as in the proposed design.

#### 9.4 High Pressure Slurry Feed Pumps

a. MLT. The CROWN CHROME triplex pump model 707 gave good service throughout. Most of the maintenance was for routine replacement of plungers and packing. Three different types of plunger were evaluated and it was found that rechromed surfaces were superior in wear resistance to either stainless steel sleeves, or new stainless steel. The potential lifetime of a chromed plunger is up to 1 000 hours and the replacement time is one hour. The pumps were modified to take rubberized chevron seals instead of the original rope packing. The normal lifetime of a chevron seal is 500 hours and the time to change a seal is one hour.

The optimum maintenance procedure is to change seals when they fail and to change the plungers only after 500 hours use except if they are obviously worn. The practice of changing plungers whenever seals are changed is wasteful.

b. SPARRO. Compared with the MLT plant, the two high pressure slurry pumps were very troublesome. However, after several plant modifications, the CROWN CHROME triplex pump model 707 gave satisfactory service in 1990. The most important points to be considered are the provision of an adequate suction head from the reactor and the prevention of calcium sulphate seed blockages in the inlet manifold.

The NATIONAL triplex pump was never truly commissioned. Although it ran well in October 1989 this performance was never repeated. The absence of a reliable stand-by pump caused a significant loss in availability. While the NATIONAL pump may have been rectified by the final overhaul, it cannot be recommended at this stage.

#### 9.5 "Twin" Versus "Single" High Pressure Feed Pumps

The twin pump system is the main difference between the two plants. The advantages claimed were:

- . maintenance costs would be reduced because a proportion of the water pressurised to 3 000 kPa would be clean and not a slurry.
- . the salinity of the combined stream at the manifold inlet could be reduced below saturation if the pretreated water was unsaturated.
- capital costs could be reduced on large plants as large scale multi-stage centrifugal pumps have a lower specific cost than positive displacement pumps.

It is true that the MONO pump ran reliably (> 99 per cent availability) on clear water and that the centrifugal pumps would probably also have done if they had been repaired. However, these claimed advantages are negated by two much more significant disadvantages.

#### a. Reactor level control

The twin pump system makes the control of the reactor level on the SPARRO plant inherently unstable, and complicates the control of the membrane pressure. These difficulties would be magnified on a large multiple unit.

The single pump system simplifies reactor level control, is reliable and is recommended for use in the design given in the separate design report.

b. Contact between pretreated water and seed

During simultaneous "back-to-back" operation of the MLT and SPARRO plants in 1990 with the same feed water, it was found that the membrane bank in the MLT plant performed better than in the SPARRO plant. The MLT plant ran for several months without any appreciable deviation from the equilibrium flux rate of  $550 \ l/m^2$ .d, whereas the SPARRO plant showed flux decline which is a sign of membrane fouling (Table 13) and would probably require cleaning on a monthly basis. The variation in the performance of the two pilot plants is attributed to the different durations of the contact time between the pretreated water and the stirred suspension before it reaches the modules. The residence time in the MLT plant was four hours whereas it was less than one minute in the SPARRO plant. This is because the pretreated water is injected by a second high pressure pump immediately upstream of the modules in the SPARRO plant instead of into the reactor. It is probable that colloidal material and other potential foulants present in

the pretreated water are removed by prolonged contact with the fine suspension of calcium sulphate particles and that they become incorporated during the process by crystallization, coprecipitation and aggregation.

The single pump system is recommended, thereby nullifying one of the claimed major advantages of the SPARRO process, i.e. reduced pumping costs as a result of the larger portion of the feed water (minewater) being pumped with a low cost pump.

The required duration of the contact between the pretreated water and seed cannot be defined on the basis of present knowledge. Four hours appears to be sufficient whereas one minute is evidently insufficient. This is a key parameter in the design of the reactor and is discussed below in Section 9.6.

#### 9.6 <u>Reactor</u>

The reactor has four purposes:

- . to induce and complete the crystallization and desupersaturation of calcium sulphate in the reject stream from the membranes
- . to remove fine colloidal material and other potential foulants from the pretreated water stream thereby preventing membrane fouling. This is effectively the final stage of pretreatment
- . to provide a constant and adequate suction head for the slurry feed pumps (P2)
- . to act as a buffer storage.

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The volume of the reactor in the SPARRO plant was sized<sup>(14)</sup> to provide one hour of residence time to complete the desupersaturation of the reject stream from the module bank. Conductivity measurements of the reject stream from the modules indicate that equilibrium is reached within several minutes and measurements taken from the recycled slurry show that desupersaturation has occurred.

As stated above (9.5), the required contact time to remove colloidal material is less than four hours. It is assumed that a residence time of one hour will be sufficient in the design of a large-scale plant.

The slurry feed pumps require a positive suction head to prevent air locks developing in the cylinders. If the level of the suspension in the reactor is maintained at 100 per cent full by admitting water on demand, then a head difference of 5 m will be adequate.

The provision of buffer storage will be met when the other criteria are met.

#### 9.7 <u>Hydrocyclone</u>

The performance of the 50 mm hydrocyclone on the MLT plant was acceptable. The water recovery could be controlled between 80 - 100 per cent by manual adjustment of the timers which divert the overflow and underflow streams either to drain or back to the reactor.

Three different hydrocyclones were installed on the SPARRO plant in 1989 - 1990 and even then the system was not satisfactory. The purpose of the design was to produce a stream high in suspended solids to make the independent discharge of seed and brine possible. Unfortunately the initial choice of a 10 mm hydrocyclone bank gave a sticky and dense underflow which caused blockages which prevented gypsum blow down. The situation was improved by the installation of a 25 mm then a 50 mm diameter hydrocyclone. The computer controlled volume measuring system was probably more sophisticated then was required. The pipework around the hydrocyclones was undersized and this made it difficult to achieve a water recovery of less than 96 per cent without excessive loss of seed which upset the calcium sulphate balance. Experience on the MLT plant shows that the hydrocyclone system can work satisfactorily but the valves and pipework must be adequately sized.

#### 10 DESIGN OF 5,78 L/s SPARRO PLANT

A detailed design and costing of a 5,78 l/s (500 kl/day) SPARRO plant was prepared. A summary of the basic design data used is given below and more detailed information is given in a separate desing report which is available on request.

#### 10.1 Pretreatment

	Quality of product water		
	рH		4 - 6
	Temperature (max)	°C	25
	Suspended solids (max)	mg/l	1
	Turbidity (max)	NTU	1
	Total chlorine (max)	mg/l	3
10.2	Reactors		
	Seed concentration	mg/l	20 - 40 000
	Seed concentration (min)	mg/l	10 000
	Residence time	h	1
10.3	Hydrocyclones		
	Water recovery	%	80 - 99
	Seed size (ave)	micron	50

## 10.4 Slurry Feed Pumps

Plunger life	h	500 - 1 000
Seal life (ave)	h	500
Membrane Banks		
Slurry velocity (ave)	m/s	2
Slurry velocity (min)	m/s	1
Flux (at 25 ° C, 4 000 kPa)	ℓ/m².d	500 - 600
Salt rejection (m/m)	%	95
Operating pressure (max)	kPa	3 000
Pass conversion (max)	%	30
Module area (each)	m <sup>2</sup>	1,75
Membrane life (min)	У	2
	Plunger life Seal life (ave) <u>Membrane Banks</u> Slurry velocity (ave) Slurry velocity (min) Flux (at 25 ° C, 4 000 kPa) Salt rejection (m/m) Operating pressure (max) Pass conversion (max) Module area (each) Membrane life (min)	Plunger lifehSeal life (ave)hMembrane BanksSlurry velocity (ave)m/sSlurry velocity (min)m/sFlux (at 25 ° C, 4 000 kPa) $l/m^2.d$ Salt rejection (m/m)%Operating pressure (max)kPaPass conversion (max)%Module area (each)m²Membrane life (min)y

## 10.6 Operating Procedures

Preliminary operating procedures and membrane cleaning and renovating procedures are included in the design report.

## 11 <u>COSTS</u>

#### 11.1 Operating Costs

Table 14 SPARRO PLANT OPERATING COSTS (1990)

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ltem	Unit Cost	Useage	Cost
Floc	1,5 g/kl	8 R/kg	1,2
Нуро	0,034 <i>l/</i> k	0,88 R/kg	3,0
Elec.	5 kWh/kl	0,091 R/kWh	45,5
Plungers*	3/500 h	R79 each	18,2
Seals *	3/500 h	R19 each	4,6
Membranes	2 years	R300 each	<u>62.0</u>
			134,5 c/kŁ

\* It is assumed in this cost estimate that the plungers and seals are changed simultaneously after 500 hours of use.

• • •

#### 11.2 Capital Costs

#### Table 15 SPARRO PLANT (5,78 1/s) CAPITAL COST ESTIMATE SUMMARY (1990)

	ltem	R000's
1.	Civil Work	151,2
2.	Process equipment	1 296,48
3.	Structural steelwork	33,7
4.	Piping and valves	170,04
5.	Electric power, wiring and distribution	28,9
6.	Instruments	142,52
7.	Painting	30,0
8.	Engineering assembly	
	Total	2 152,8
	Contingency (20 %)	430.56
	Total	<u>2 583,36</u>

\* Plant is designed for a 20 year life.

#### 12 FURTHER RESEARCH

While it is considered that the design given in the separate design report is realistic and viable, certain areas require optimization. This may be carried out when a commercial plant is constructed, by application of work done on other projects (e.g. by ESCOM), or by small scale laboratory studies. The areas of future research are in the following fields:

#### a. Reactor

The required contact time between the seed slurry and pretreated water has not been quantified. It is known that four hours is sufficient but this may be excessive. The type of reactions which occur in the reactor and their kinetics need to be determined.

#### b. Manifolding

The delivery of slurry from a single pump to a number of separate module banks and the detailed design of the inlet manifolds require further attention. Problems of this kind have been experienced by ESCOM and the University of Natal have expertise in this field<sup>(13)</sup>.

c. Hydrocyclone

A laboratory scale evaluation of various sizes of hydrocyclone operating with calcium sulphate slurries would provide valuable information. The behaviour of the cyclone could be correlated with a computer model<sup>(12)</sup>. This work does not need to be carried out on an operating plant.

#### 13 POTENTIAL FOR APPLICATION

The most likely areas of application at this stage appear to be in the field of effluent treatment. Possible applications are in gold mining, coal mining, electricity generation, petroleum refining and the steel industry.

The widespread use of slurry reverse osmosis in the gold mining industry as a means of generating potable water or as a means of upgrading service water for hydro-power is unlikely in the near future.

## 14 <u>CONCLUSIONS</u>

- 1. The slurry reverse osmosis desalination process is technically viable for use with calcium sulphate scaling mine water.
- 2. In order to maintain an acceptable membrane life, mine water must be pretreated to pH 4 6, temperature < 25 °C, total suspended solids < 1 mg/*l*, and chlorinated.
- 3. It is not necessary to remove iron and manganese in the pretreatment stage.
- 4. A membrane life of at least two years is predicted provided specified operating procedures are maintained.
- 5. The corrected membrane flux on the MLT plant, at equilibrium, correlated closely with the predicted manufacturer's design value of 0,0064  $l/m^2$ .s (550  $l/m^2$ .d) at 25 °C and 4 000 kPa.
- 6. The membrane flux on the SPARRO plant declined below the design value, probably because of membrane degradation. It could be restored by cleaning with citric acid and Biotex solution to give a membrane life of two years.
- 7. The straight-through module bank (MLT) is preferred to the tapered bank, at the lower pass conversion of 30 per cent.
- 8. The single slurry feed pump system (MLT) is superior to the twin feed pump system (SPARRO) in terms of reactor level control, and membrane fouling.
- 9. The Crown Chrome triplex pump performed satisfactorily as a slurry feed pump.

- 10. A water recovery of 85 95 per cent was achieved.
- 11. A salt rejection of 90 95 per cent (wrt feed) and 75 per cent (overall) was achieved.
- 12. The product water was suitable for human consumption with respect to salinity.
- 13. The operating cost of a slurry reverse osmosis desalination plant is estimated to be R1,35/k<sup>2</sup> excluding capital charges (1990).
- The capital cost of a slurry reverse osmosis plant to produce 5,78 l/s (500 kl/d) at a water recovery of 90 per cent is estimated to be R2,58 million in 1990 terms, as designed for a 20 year life.

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## APPENDIX I ANALYSIS OF PRETREATMENT, MLT, AND SPARRO PLANTS AVAILABILITIES FROM JULY 1989 TO AUGUST 1990

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## APPENDIX I

## ANALYSIS OF MLT AND SPARRO PLANT AVAILABILITIES JULY 1989 TO AUGUST 1990

## SUMMARY OF MLT PLANT OPERATING TIMES AND AVAILABILITIES

		Plant Run	Shutdown		Max Possible	Availability
MLT			Not Planned	Planned		
		hrs	hrs	hrs	hrs	%
March April May June July August September October November December January February March April May June July August	1989 1989 1989 1989 1989 1989 1989 1989	Comm Comm Comm 656 360 207 614 562 345 291 620 681 501 582 370 576 563	88 384 297 130 158 135 36 52 63 104 114 254 168 133	0 0 216 0 264 417 0 115 48 96 0 48	744 720 744 720 744 720 744 720 744 720 744 720 744 720 744 720 744 720 744 720	Comm Comm Comm 88 48 41 83 78 72 89 92 92 83 84 59 77 81
					Ave:	76

		Plant Run	Shutdown		Max Possible	Availability
SPARRO			Not Planned	Planned		
		hrs	hrs	hrs	hrs	%
February March April May June July August September October November December January February March April May June July August	1989 1989 1989 1989 1989 1989 1989 1989	Comm Comm Comm Comm 344 348 370 638 454 194 60 450 517 442 595 528 314 410	400 396 134 106 266 118 10 222 227 163 101 96 430 286	0 216 0 432 674 0 115 48 96 0 48	672 744 720 744 720 744 744 720 744 720 744 744 672 744 720 744 720 744 720 744	Comm Comm Comm Comm 46 47 73 86 63 62 86 63 62 86 67 69 73 85 84 42 59
					Ave:	67

## SUMMARY OF SPARRO OPERATING TIMES AND AVAILABILITIES

.

		Shutdown	Max Possible	Availability
PRETREATMENT		Not Planned		
		hrs	hrs	%
March April May June July August September October November December January February March April May June July August	1989 1989 1989 1989 1989 1989 1989 1989	85 336 96 42 5 38 0 16 0 0 0 0 0 0	744 720 744 720 744 720 744 720 744 720 744 720 744 720 744 720 744 720 744	- - - - - - - - - - - - - - - - - - -
			Ave:	93

SUMMARY OF PRETREATMENT PLANT OPERATING TIMES AND AVAILABILITIES

Ψ.

## ANALYSIS OF UNPLANNED STOPPAGES JULY 1989

Plant	Off-Line Time (Hours)
Pretreatment Operator absent Air shortage Delivery pump No 2 Clarifier pump Clarifier Total	29 5 24 3 24 85
<u>MLT Plant</u> Operator absent Pretreatment plant breakdown Slurry feed pump Total	29 56 3 88
<u>SPARRO Plant</u> Operator absent Pretreatment breakdown Mine water pump P1A Slurry recycle pumps P2A, B Other Total	29 56 100 200 15 400

## ANALYSIS OF UNPLANNED STOPPAGES AUGUST 1989

Plant	Off-Line Time (Hours)
Pretreatment Operator absent Dosing pump Delivery pump No 2 Total	96 168 72 336
<u>MLT Plant</u> Operator absent Pretreatment Slurry feed pump Total	96 240 48 384
<u>SPARRO Plant</u> Operator absent Pretreatment Slurry recycle pumps Total	96 240 60 396

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## STOPPAGES ALLOCATED TO PREDOMINANT CAUSE

## ANALYSIS OF UNPLANNED STOPPAGES SEPTEMBER 1989

Plant	Off-Line Time (Hours)
Pretreatment Delivery pump No 2 Caustic soda dosing Total	48 48 96
<u>MLT Plant</u> Pretreatment breakdown Reject flow control Total	96 201 297
<u>SPARRO Plant</u> Pretreatment breakdown Bursting discs Pump P2A, P2B Total	96 12 26 134

## ANALYSIS OF UNPLANNED STOPPAGES OCTOBER 1989

Plant	Off-Line Time (Hours)
Pretreatment Water shortage Floc dosing Delivery pump No 2 Total	8 10 24 42
<u>MLT Plant</u> Pretreatment plant breakdown Slurry feed pump Modules Total	42 32 56 130
<u>SPARRO Plant</u> Pretreatment breakdown Slurry recycle pumps Total	42 64 106

## STOPPAGES ALLOCATED TO PREDOMINANT CAUSE

## ANALYSIS OF UNPLANNED STOPPAGES NOVEMBER 1989

Plant	Off-Line Time (Hours)
Pretreatment pH control Total	5 5
<u>MLT Plant</u> Pretreatment plant breakdown Bursting disc Power failure Slurry feed pump Total	5 5 8 140 158
<u>SPARRO Plant</u> Pretreatment breakdown Bursting disc Power failure Slurry feed pump Total	5 5 8 248 266

## ANALYSIS OF UNPLANNED STOPPAGES DECEMBER 1989

Plant	Off-Line Time (Hours)
Pretreatment pH control Operators absent Total	28 10 38
<u>MLT Plant</u> Pretreatment plant breakdown Operators absent Modules Slurry feed pump Total	28 10 1 96 135
<u>SPARRO Plant</u> Pretreatment breakdown Slurry feed pumps Total	28 90 118

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## STOPPAGES ALLOCATED TO PREDOMINANT CAUSE

## ANALYSIS OF UNPLANNED STOPPAGES JANUARY 1990

Plant	Off-Line Time (Hours)
MLT Plant Power failure Operator absent Slurry feed pump Instrumentation Total	15 12 4 5 36
SPARRO Plant Instrumentation Slurry recycle pumps Total	6 4 10

## ANALYSIS OF UNPLANNED STOPPAGES FEBRUARY 1990

Plant	Off-Line Time (Hours)
Pretreatment pH meter	16
<u>MLT Plant</u> Pretreatment plant breakdown Slurry feed pump Modules failures, changes Total off-line	16 25 11 52
SPARRO Plant Pretreatment Generator Feed pump P1 Slurry recycle pumps P2 Modules Reject line blockage Total	16 8 48 25 83 42 222

## STOPPAGES ALLOCATED TO PREDOMINANT CAUSE

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## ANALYSIS OF UNPLANNED STOPPAGES MARCH 1990

Plant	Off-Line Time (Hours)
<u>MLT Plant</u> Slurry feed pump Modules failure, changes Total	49 14 63
<u>SPARRO Plant</u> Air pressure ex mine/mods Slurry recycle pumps Module failure, changes Water meter installation Total	49 150 20 8 227

## ANALYSIS OF UNPLANNED STOPPAGES APRIL 1990

Plant	Off-Line Time (Hours)
<u>MLT</u> Slurry feed pump Reactor Power failure Total	90 8 6 104
<u>SPARRO</u> Valves - Iow gas pressure Slurry feed pump plungers Power failure Module change Slurry feed pump electric overload Total	11 31 2 10 109 163

## STOPPAGES ALLOCATED TO PREDOMINANT CAUSE

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# ANALYSIS OF UNPLANNED STOPPAGES MAY 1990

Plant	Off-Line Time (Hours)
MLT Module burst Slurry feed pump Total	5 109 114
<u>SPARRO</u> Slurry feed pump Valves - Iow gas pressure Power failure Total	81 5 15 101

## ANALYSIS OF UNPLANNED STOPPAGES JUNE 1990

Plant	Off-Line Time (Hours)
<u>MLT</u> Slurry feed pump Total	254 254
<u>SPARRO</u> Mono pump P1 Module cleaning Instrumentation Total	26 49 21 96

### STOPPAGES ALLOCATED TO PREDOMINANT CAUSE

## ANALYSIS OF UNPLANNED STOPPAGES JULY 1990

Plant	Off-Line Time (Hours)
<u>MLT</u> Slurry feed pump Reactor agitator Instrumentation Total	42 120 6 168
SPARRO Module renovation Instrumentation Power failure Slurry feed pump Operator absent Total	336 6 1 70 17 430

## ANALYSIS OF UNPLANNED STOPPAGES AUGUST 1990

Plant	Off-Line Time (Hours)
<u>MLT</u> Reactor agitator Operator absent Power failure Total	88 15 30 133
<u>SPARRO</u> Module renovation Slurry feed pump Operator absent Total	192 79 15 286

## STOPPAGES ALLOCATED TO PREDOMINANT CAUSE

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# APPENDIX II <u>GRAPHS OF PRETREATMENT, MLT AND SPARRO PLANTS</u> <u>PERFORMANCE FROM FEBRUARY 1989 TO AUGUST 1990</u>

#### APPENDIX II

#### **GRAPHS**

- 1. Raw mine water conductivity
- 2. Clarifier overflow pH
- 3. Filter outlet pH
- 4. Filter outlet turbidity
- 5. MLT feed conductivity
- 6. MLT feed total suspended solids
- 7. MLT feed pH
- 8. MLT product conductivity
- 9. MLT rejection (wrt MLT feed)
- 10. MLT corrected flux
- 11. SPARRO feed conductivity
- 12. SPARRO feed total suspended solids
- 13. SPARRO feed pH
- 14. SPARRO product conductivity
- 15. SPARRO rejection (wrt SPARRO feed)
- 16. SPARRO corrected flux

Graph A - 1989 elapsed hours

Graph B - 1990 elapsed hours

1 000 hours equivalent to 41,66 calendar days

31 days equivalent to 744 hours

Off-line time is shown by gap in graph.

#### APPENDIX II

#### **GRAPHS**

- 1. Raw mine water conductivity
- 2. Clarifier overflow pH
- 3. Filter outlet pH
- 4. Filter outlet turbidity
- 5. MLT feed conductivity
- 6. MLT feed total suspended solids
- 7. MLT feed pH
- 8. MLT product conductivity
- 9. MLT rejection (wrt MLT feed)
- 10. MLT corrected flux
- 11. SPARRO feed conductivity
- 12. SPARRO feed total suspended solids
- 13. SPARRO feed pH
- 14. SPARRO product conductivity
- 15. SPARRO rejection (wrt SPARRO feed)
- 16. SPARRO corrected flux

Graph A - 1989 elapsed hours Graph B - 1990 elapsed hours

1 000 hours equivalent to 41,66 calendar days

31 days equivalent to 744 hours

Off-line time is shown by gap in graph.



GRAPH 1A : Raw Mine Water Conductivity - 1989



# GRAPH 1B : Raw Mine Water Conductivity - 1990



GRAPH 2A : Clarifier Overflow pH - 1989

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GRAPH 2B : Clarifier Overflow pH - 1990

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GRAPH 3A : Filter Outlet pH - 1989

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GRAPH 3B : Filter Outlet pH - 1990





ω ω elapsed time (hours) x  $10^3$ 2 Ο Т Ю T ນ I 1 Ì l ດ ω ~ ω 2 4 Ο (UTU) turbidity (NTU)

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GRAPH 4B : Filter Outlet Turbidity - 1990



GRAPH 5A : MLT Feed Conductivity - 1989





# GRAPH 6A : MLT Feed Total Suspended Solids - 1989



GRAPH 6B : MLT Feed Total Suspended Solids - 1990

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GRAPH 7A : MLT Feed pH - 1989

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GRAPH 7B : MLT Feed pH - 1990



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GRAPH 8A : MLT Product Conductivity - 1989

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GRAPH 8B : MLT Product Conductivity - 1990



GRAPH 9A : MLT Rejection (wrt MLT Feed) - 1989



GRAPH 9B : MLT Rejection (wrt MLT Feed) - 1990



GRAPH 10A : MLT Corrected Flux - 1989

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GRAPH 10B : MLT Corrected Flux - 1990



# GRAPH 11A : SPARRO Feed Conductivity - 1989



GRAPH 11B : SPARRO Feed Conductivity - 1990



GRAPH 12A : SPARRO Feed Total Suspended Solids - 1989

ω G elapsed time (hours) x 10<sup>3</sup> 2 Ο ł 32 30 28 26 24 22 20 **8** 16 5 ω 4 5 ω Ο 2 4

total suspended solids (mg/ $\lambda$ ) x 10<sup>3</sup>

GRAPH 12B : SPARRO Feed Total Suspended Solids - 1990



GRAPH 13A : SPARRO Feed pH - 1989

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GRAPH 13B : SPARRO Feed pH - 1990



GRAPH 14A : SPARRO Product Conductivity - 1989



GRAPH 14B : SPARRO Product Conductivity - 1990



GRAPH 15A : SPARRO Rejection (wrt SPARRO Feed) - 1989



GRAPH 15B : SPARRO Rejection (wrt SPARRO Feed) - 1990



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GRAPH 16A : SPARRO Corrected Flux - 1989

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GRAPH 16B : SPARRO Corrected Flux - 1990

## APPENDIX III REGISTER OF MODULES AND SALT REJECTIONS OF MODULES INSTALLED ON MLT PLANT FROM MARCH 1989 TO AUGUST 1990

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## APPENDIX III

#### MLT PLANT MODULE : REGISTER

## POSITION NO. 1

DATE	PLANT	MODULE	R COND.	HOURS IN	FLUX
	HOURS	NO.	%	USE	<i>l</i> /m <sup>2</sup> .d
06.03.1989 01.06.1989 01.06.1989 22.06.1989 28.07.1989 13.11.1989 20.11.1989 20.11.1989 30.11.1989 15.12.1989 15.12.1989 15.12.1989 15.12.1989 22.12.1989 19.01.1990 31.01.1990 02.02.1990 13.02.1990 13.02.1990 12.03.1990 12.03.1990 11.04.1990 19.06.1990 19.06.1990	0 1047 1047 1472 2190 3070 3686 3829 4003 4104 4218 4280 4370 4386 4592 4638 4638 4638 4678 4638 5049 5531 5823 6190 7290 7456	881324 890034 890034 890034 890034 890034 890034 890034 890034 890034 890034 890034 890034 890034 890034 890034 890034 890034 890034 890034 891035 891035 891035 891035 891035 891035 891035 891035 891035 891035 891035 891035	- 98 97 97 83 79,6 74,5 74,5 77,3 75,1 77,6 84,2 98,1 94,47 94,75 94,39 93,80	INSTALL 0 0 425 1143 2023 2639 2782 2956 3057 3171 3233 3323 3339 3545 INSTALL 0 40 270 411 893 1185 1552 2652 2818	400
13.07.1990	7636	891035	94,2	2998	491
30.08.1990	8554	891035	91,3	3916	

#### POSITION NO. 2

DATE	PLANT HOURS	MODULE NO.	R COND. %	HOURS IN USE	FLUX <i>L</i> /m <sup>2</sup> .d
06.03.1989 01.06.1989 22.06.1989 28.07.1989 12.10.1989 13.11.1989 30.11.1989 30.11.1989 30.11.1989 15.11.1989 15.11.1989 15.11.1989 19.01.1990 31.01.1990 31.01.1990 02.02.1990 19.02.1990 19.02.1990 19.02.1990 11.04.1990 11.04.1990	0 1047 1472 2190 3070 3686 3829 4003 4104 4218 4280 4370 4386 4592 4638 4678 4592 4638 4678 4592 5049 5531 5823 6190	881343 88	- 90 87 86,5 62 59,3 51,6 48,7 50,6 46 43,3 47,5 46,2 54,3 97,6 94,1 95,45 94,6	INSTALL 0 1047 1472 2190 3070 3686 3829 4003 4104 4218 4280 4370 4386 4592 INSTALL 0 40 270 411 893 1185 1552 2652	542
19.06.1990 05.07.1990 13.07.1990 30.08.1990	7290 7456 7636 8554	891062 891062 891062 891062	96,25 94,2 95,1	2052 2818 2998 3916	542 653

POSITION	Position of module in stack 1 - 20
PLANT HOURS	Metered plant running time
MODULE NO.	Membratek serial number of module
R(COND)%	Salt rejection based on conductivity measurement
HOURS IN USE	Operating time elapsed since time of installation
FLUX	Measured flux in $l/m^2/.d$ corrected to 25 ° C and 4 000 kPa.

DATE	PLANT	MODULE	R COND.	HOURS IN	FLUX
	HOURS	NO.	%	USE	<i>L</i> /m <sup>2</sup> .d
06.03.1989 01.06.1989 22.06.1989 28.07.1989 12.10.1989 12.10.1989 13.11.1989 20.11.1989 30.11.1989 04.12.1989 11.12.1989 15.12.1989 15.12.1989 19.01.1990 22.02.1990 13.02.1990 13.02.1990 13.02.1990 12.03.1990 21.04.1990 11.04.1990	0 1047 1472 2190 3070 3686 3829 4003 4104 4218 4280 4370 4386 4592 4678 4908 5049 5531 5823 6190 7290	881094 881094 881094 881094 881510	- 87 85 83 57 - 88,3 83,9 67,4 88,8 86,2 84,8 86,6 90,9 91,3 85,3 86,67 85,3 85,3 84,02	INSTALL 0 1047 1472 2190 3070 INSTALL 0 616 759 933 1034 1148 1210 1300 1316 1522 1608 1838 1838 1979 2461 2753 3120 4220	900
05.07.1990	7456	881510	90,02	4386	674
13.07.1990	7636	881510	84,3	4566	
30.08.1990	8554	881510	86,6	5484	

#### **POSITION NO. 4**

DATE	PLANT	MODULE	R COND.	HOURS IN	FLUX
	HOURS	NO.	%	USE	北/m <sup>2</sup> .d
06.03.1989 01.06.1989 22.06.1989 28.07.1989 12.10.1989 13.11.1989 20.11.1989 20.11.1989 04.12.1989 15.12.1989 15.12.1989 15.12.1989 22.12.1989 19.01.1990 29.01.1990 31.01.1990 19.02.1990 19.02.1990 19.02.1990 11.04.1990 11.04.1990 11.06.1990	0 1047 1472 2190 3070 3686 3829 4003 4104 4218 4280 4370 4386 4592 4638 4592 4638 4592 4638 4592 4638 5049 5531 5823 6190 7044 7290	NO. 881321 891072 8	% 91 86 85 64 54,3 51,6 56,8 49,9 46,2 42,8 45,7 48,6 54,1 97,1 97,1 94,68 95,15 94,57 97,07	USE INSTALL 0 1047 1472 2190 3070 3686 3829 4003 4104 4218 4280 4370 4386 4592 INSTALL 0 270 411 893 1185 1552 INSTALL 0 246	<i>1</i> /m².a
05.07.1990	7456	900095	98,38	412	719
13.07.1990	7636	900095	97,5	592	
30.08.1990	8554	900095	96,8	1510	

DATE	PLANT HOURS	MODULE NO.	R COND. %	HOURS IN USE	FLUX <i>t</i> /m <sup>2</sup> .d
06.03.1989 01.06.1989 02.06.1989 22.06.1989 25.09.1989 12.10.1989 13.11.1989 30.11.1989 30.11.1989 04.12.1989 15.12.1989 15.12.1989 19.01.1990 02.02.1990 13.02.1990 12.03.1990 12.03.1990 11.04.1990	0 1047 1061 1472 2190 2751 3070 3686 3829 4003 4104 4218 4280 4370 4386 4370 4386 4592 4678 4592 4678 4908 5049 5531 5823 6190	881320 881320 890032 890032 890032 881492	2 91 96 96 93 88,6 86,7 86,5 88,7 86,5 88,7 86,9 86,7 88,6 87,8 92,3 92,2 89,42 90,13 89,22	INSTALL 0 1047 INSTALL 0 411 1129 INSTALL 0 319 935 1078 1252 1353 1467 1529 1619 1635 1841 1927 2157 2298 2780 3072 3439	7.11.0
19.06.1990 05.07.1990 13.07.1990 30.08.1990	7290 7456 7636 8554	881492 881492 881492 881492	88,61 92,32 88,05 90,0	4539 4705 4885 5800	623 622

## POSITION NO. 6

DATE	PLANT HOURS	MODULE NO.	R COND. %	HOURS IN USE	FLUX <i>l</i> /m <sup>2</sup> .d
06.03.1989 01.06.1989 22.06.1989 28.07.1989 12.10.1989 13.11.1989 20.11.1989 30.11.1989 04.12.1989 15.12.1989 15.12.1989 19.01.1990 29.01.1990 29.01.1990 02.02.1990 13.02.1990 19.02.1990 12.03.1990	HOURS 0 1047 1472 2190 3070 3686 3829 4003 4104 4218 4280 4370 4386 4592 4638 4678 4838 4678 4838 4678 5049 5531	NO. 881351 890107 891081 891081 891081 891081 891081 891081 891081	% 91 90 88 63,5 58,6 51,2 65,2 51 46,2 44,2 44,2 44,5 46,9 57,8 97,4	USE INSTALL 0 1047 1472 2190 3070 3686 3829 4003 4104 4218 4280 4370 4386 4592 INSTALL 0 40 INSTALL 0 70 211 693	ℓ/m².d
12.03.1990 26.03.1990 11.04.1990	5531 5823 6190	891081 891081 891081	94,47 94,90 94,56	693 985 1352	
04.05.1990 19.06.1990 05.07.1990 13.07.1990	6504 7290 7456 7636	900084 900084 900084 900084	97,12 98,33 97,03	INSTALL 0 786 952 1132	730
30.08.1990	8554	900084	97,0	2050	625

06.03.1989 0 881344 - INSTALL 0   01.06.1989 1047 881344 90 1047   22.06.1989 1472 881344 88 1472   28.07.1989 2190 881344 86,5 2190   12.10.1989 3070 881344 65,5 3070   13.11.1989 3686 881344 50,2 3686   20.11.1989 3629 881344 50,7 3829   30.11.1989 4003 881344 50,8 4104   11.12.1989 4104 881344 46,4 4218   15.12.1989 4280 881344 46,2 4386   22.12.1989 4370 881344 46,2 4386   29.01.1990 4592 881344 46,2 4386   29.01.1990 4592 881344 46,2 4386   29.01.1990 4592 881344 46,2 4386   29.01.1990 4588 891104 - INSTALL 0	DATE	PLANT HOURS	MODULE NO.	R COND. %	HOURS IN USE	FLUX <i>l</i> /m <sup>2</sup> .d
05.07.1990 7456 891104 93,95 2818 13.07.1990 7656 891104 93.9	06.03.1989 01.06.1989 22.06.1989 28.07.1989 12.10.1989 13.11.1989 30.11.1989 30.11.1989 04.12.1989 15.12.1989 15.12.1989 15.12.1989 19.01.1990 31.01.1990 31.01.1990 02.02.1990 13.02.1990 12.03.1990 12.03.1990 11.04.1990 11.04.1990	0 1047 1472 2190 3070 3686 3829 4003 4104 4218 4280 4370 4386 4592 4638 4578 4592 4638 4578 4598 5049 5531 5823 6190 7290	881344 881344 881344 881344 881344 881344 881344 881344 881344 881344 881344 881344 881344 881344 881344 881344 891104 891104 891104 891104 891104 891104	90 88 86,5 56,7 50,9 56,4 44,7 85,2 50,8 46,4 49,2 5 ,2 84,9 46,9 94,2 94,433 6 84,336	INSTALL 0 1047 1472 2190 3070 3686 3829 4003 4104 4218 4280 4370 4386 4592 INSTALL 0 40 270 411 893 1185 1552 2652	702
	05.07.1990 13.07.1990	7456 7636	891104 891104 891104	95,95 93,9 95 2	2052 2818 2998	520

#### POSITION NO. 8

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DATE	PLANT HOURS	MODULE NO.	R COND. %	HOURS IN USE	FLUX <i>l</i> /m <sup>2</sup> .d
06.03.1989 01.06.1989 22.06.1989 28.07.1989 13.11.1989 20.11.1989 20.11.1989 20.11.1989 04.12.1989 10.12.1989 11.12.1989 15.12.1989 15.12.1989 22.2.2.1989 19.01.1990 02.02.1990 13.02.1990 19.02.1990 12.03.1990 26.03.1990	HOURS 0 1047 1472 2190 3070 3686 3829 4003 4104 4189 4218 4280 4370 4386 4370 4386 4592 4678 4908 5049 5531 5823 5823	NO. 881348 881348 881348 881348 881348 881348 881348 881348 881348 881348 890017 890017 890017 890017 890017 890017 890017 890017 890017 890017	% 88 87 85 62.5 52.8 48,7 49,8 93,2 94,2 94,2 92,9 92,6 94,9 89,44 90,42	USE INSTALL 0 1047 2190 3070 3686 3829 4003 4104 INSTALL 0 29 91 181 197 403 489 719 860 1342 1634	<i>t/</i> m².d
19.06.1990 05.07.1990 13.07.1000	7290 7456 7636	890017 890017	89,94 92,66	3101 3267 2447	623
30.08.1990	8554	890017	90,8	4365	686

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DATE	PLANT	MODULE	R COND.	HOURS IN	FLUX
	HOURS	NO.	%	USE	<i>l</i> /m <sup>2</sup> .d
06.03.1989 01.06.1989 22.06.1989 28.07.1989 12.10.1989 13.11.1989 30.11.1989 30.11.1989 04.12.1989 15.12.1989 15.12.1989 15.12.1989 15.01.1990 31.01.1990 02.02.1990 13.02.1990 12.03.1990 12.03.1990 11.04.1990 11.04.1990	0 1047 1472 2190 3070 3686 3829 4003 4104 4218 4280 4370 4386 4592 4638 4638 4638 4678 4909 5531 5823 6190	881298 891047 891047 891047 891047 891047	- 88 85 62,5 61 52,2 53,8 62,1 52,2 53,8 62,1 52,2 53,8 62,1 52,2 53,8 62,2 44,2,4 42,2 44,4 47,6 1 98,5 94,3 7 95,11 94,3 7	INSTALL 0 1047 1472 2190 3070 3686 3829 4003 4104 4218 4280 4370 4386 4592 INSTALL 0 40 270 411 893 1185 1552 2652	604
05.07.1990	7456	891047	96,21	2818	
13.07.1990	7636	891047	94,05	2998	
30.08.1990	8554	891047	94,0	3916	565

#### **POSITION NO. 10**

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DATE	PLANT	MODULE	R COND.	HOURS IN	FLUX
	HOURS	NO.	%	USE	<i>L</i> /m <sup>2</sup> .d
06.03.1989 01.06.1989 22.06.1989 20.07.1989 28.07.1989 13.11.1989 30.11.1989 30.11.1989 30.11.1989 15.12.1989 15.12.1989 15.12.1989 19.01.1990 02.02.1990 13.02.1990 13.02.1990 12.03.1990 11.04.1990 30.04.1990	0 1047 1472 1800(est) 2190 3070 3686 3829 4003 4104 4218 4280 4370 4386 4592 4678 4392 4678 4908 5049 5531 5823 6190 6458 7280	881295 881295 881295 890016	* 88 76,5 96 93 80,8 78,3 77,9 83,1 77,9 83,1 79,4 77,7 80,5 79,9 85,1 86,5 85,1 86,5 83,09 83,83 83,48 96 66	INSTALL 0 1047 1472 INSTALL 0 390 1270 1886 2029 2203 2304 2418 2480 2570 2586 2792 2878 3108 3249 3731 4023 4390 INSTALL 0 822	656
19.06.1990	7290	900093	96,66	832	833
05.07.1990	7456	900093	97,95	998	
13.07.1990	7636	900093	96,9	1178	
30.08.1990	8554	900093	96,7	2096	

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DATE	PLANT HOURS	MODULE NO.	R COND. %	HOURS IN USE	FLUX <i>t</i> /m <sup>2</sup> .d
06.03.1989 01.06.1989 22.06.1989 28.07.1989 12.10.1989 13.11.1989 20.11.1989 20.11.1989 20.11.1989 20.11.1989 20.11.1989 20.12.1989 15.12.1989 15.12.1989 15.12.1989 22.12.1989 15.12.1989 15.01.1990 29.01.1990 13.02.1990 13.02.1990 12.03.1990 26.03.1990 11.04.1990 11.04.1990	HOURS 0 1047 1472 2190 3070 3686 3727 3829 3954 4003 4104 4218 4280 4370 4386 4592 4638 4678 4908 5049 5531 5823 6190	NO. 881300 881300 881300 881300 881300 881300 890445 890445 890445 890445 881451 891097 8	% 91 86,5 86,5 61 51,1 91,9 42,8 31,9 27,6 25,5 29,3 27,5 37,3 97,5 93,76 94,65 94,12	USE INSTALL 0 1047 1472 2190 3070 3686 INSTALL 0 102 (ex. 12) 49 150 264 326 416 432 638 INSTALL 0 40 270 411 893 1185 1552	ℓ/m².d
19.06.1990 05.07.1990 13.07.1990	7290 7456 7636	891097 891097 891097	94,18 96,10 94 25	2052 2818 2088	228
30.08.1990	8554	891097	95,8	3916	555

#### POSITION NO. 12

DATE	PLANT HOURS	MODULE NO.	R COND. %	HOURS IN USE	FLUX <i>L</i> /m <sup>2</sup> .d
06.03.1989 01.06.1989 22.06.1989 28.07.1989 13.11.1989 15.11.1989 15.11.1989 30.11.1989 30.11.1989 15.12.1989 15.12.1989 15.12.1989 19.01.1990 02.02.1990 13.02.1990 13.02.1990 19.02.1990 19.02.1990 19.02.1990 19.02.1990 11.04.1990 19.06.1990 05.07.1990	0 1047 1472 2190 3070 3686 3727 3829 4003 4104 4218 4280 4370 4386 4592 4678 4908 4929 5049 5531 5823 6190 7290 7456	NU. 881451 881451 881451 881451 881451 880376 891094 891094 891094 891094	* 84 80 79 53 43,2 91,9 88,9 90,2 87,2 86,1 88,7 91,5 91,5 91,5 94,4 95,25 95,70 95,13 94,80 96,77	USE INSTALL 0 1047 1472 2190 3070 3686 INSTALL 0 102 276 377 491 553 643 659 865 951 1181 INSTALL 0 120 602 894 1261 2361 2527	£/m².d
13.07.1990 20.07.1990 30.08.1990	7636 7805 8554	891094 900098 900098	95,00 97,8	2707 INSTALL 0 749	719

DATE	PLANT	MODULE	R COND.	HOURS IN	FLUX
	HOURS	NO.	%	USE	<i>l</i> /m <sup>2</sup> .d
06.03.1989 01.06.1989 22.06.1989 28.07.1989 12.10.1989 13.11.1989 15.11.1989 20.11.1989 20.11.1989 04.12.1989 15.12.1989 15.12.1989 15.12.1989 15.12.1989 15.01.1990 02.02.1990 13.02.1990 01.03.1990 12.03.1990 12.03.1990 11.04.1990 11.04.1990	0 1047 1472 2190 3070 3686 3727 3829 4003 4104 4218 4280 4370 4386 4592 4678 4592 4678 4908 5049 5276 5531 5823 6190 7290	881299 881299 881299 881299 881299 880379	- 86 85 83 57 47,1 94 89,4 90,8 87,8 86,9 88,8 87,8 92,1 91,5 - 96,58 96,58 96,58 96,58 96,58	INSTALL 0 1047 1472 2190 3070 3686 INSTALL 0 102 276 377 491 553 643 659 865 951 1181 1322 INSTALL 0 255 547 914 2014	579
05.07.1990	7456	900020	96,95	2180	674
13.07.1990	7636	900020	96,1	2360	
30.08.1990	8554	900020	96,1	3278	

#### **POSITION NO. 14**

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DATE	PLANT	MODULE	R COND.	HOURS IN	FLUX
	HOURS	NO.	%	USE	<i>l</i> /m <sup>2</sup> .d
01.03.1989 01.06.1989 22.06.1989 28.07.1989 12.12.1989 13.11.1989 20.11.1989 20.11.1989 20.11.1989 04.12.1989 15.12.1989 15.12.1989 15.12.1989 22.12.1989 15.12.1989 22.12.1989 19.01.1990 02.02.1990 19.02.1990 19.02.1990 19.02.1990 19.02.1990 11.03.1990 26.03.1990 11.04.1990 19.06.1990	0 1047 1472 2190 3070 3686 3727 3829 4003 4104 4218 4280 4370 4386 4392 4678 4392 4678 4908 5049 5276 5531 5823 6190 7290	881103 881103 881103 881103 881103 881103 881103 890686 8900686 890023 900023 900023	91 89 87 69 61,1 91,6 90,6 92 90,4 83,7 90,9 90,5 93,7 93,1 96,15 96,65 96,63 96,03	INSTALL 0 1047 2190 3070 3686 INSTALL 0 102 276 377 491 553 643 659 865 951 1181 1322 INSTALL 0 255 547 914 2014	675
05.07.1990	7456	900023	97,20	2180	684
13.07.1990	7636	900023	95,9	2360	
30.08.1990	8554	900023	96,5	3278	

DATE	PLANT	MODULE	R COND.	HOURS IN	FLUX
	HOURS	NO.	%	USE	<i>≹</i> /m <sup>2</sup> .d
06.03.1989 01.06.1989 22.06.1989 28.07.1989 28.07.1989 12.10.1989 13.11.1989 15.11.1989 20.11.1989 20.11.1989 20.11.1989 20.11.1989 20.11.1989 21.12.1989 15.12.1989 15.12.1989 15.12.1989 15.12.1989 19.01.1990 22.02.1990 13.02.1990 13.02.1990 13.02.1990 11.04.1990 21.04.1990 21.04.1990 22.04.1990	HOURS 0 1047 1472 1800 2190 2756 3070 3686 3727 3829 4003 4104 4218 4280 4370 4386 4592 4678 4908 5049 5531 5823 6190 6303	NO. 881502 881502 890031 890031 890031 881108 881108 881108 890685 8	% 80 75 98 93,5 87,8 89,8 89,6 91,3 89,6 91,3 89,6 91,3 89,6 91,3 89,6 91,3 89,6 91,3 89,6 91,3 89,6 91,3 89,6 91,3 89,5 90,7 90,1 93,3 92,2	USE INSTALL 0 1047 INSTALL 0 390 INSTALL 0 314 930 INSTALL 0 102 276 377 491 553 643 659 865 951 1181 1322 1864 2096 2463 INSTALL 0 INSTALL 0 INSTALL 0	<i>l</i> /m².d
19.06.1990	7290	900096	96,72	987	743
05.07.1990	7456	900096	97,82	1153	
13.07.1990	7636	900096	96,0	1333	
30.08.1990	8554	900096	96,5	2251	

## POSITION NO. 16

DATE	PLANT	MODULE	R COND.	HOURS IN	FLUX
	HOURS	NO.	%	USE	<i>L</i> /m <sup>2</sup> .d
06.03.1989 01.06.1989 22.06.1989 28.07.1989 12.10.1989 13.11.1989 20.11.1989 20.11.1989 30.11.1989 04.12.1989 15.12.1989 15.12.1989 22.12.1989 19.01.1990 31.01.1990 31.01.1990 02.02.1990 13.02.1990 13.02.1990 13.02.1990 13.02.1990 11.04.1990 21.04.1990	0 1047 1472 2190 3070 3686 3829 4003 4104 4218 4280 4370 4386 4592 4638 4655 4678 4908 5049 5531 5823 6190	881309 881200 881200 8818000000000000000000000	92,5 90 88 69 57,5 53,7 56,4 53,6 49,1 48,9 51,2 49,2 58,8 51,2 49,2 58,8 96,9 23,05 93,53 92,89	INSTALL 0 1047 1472 2190 3070 3686 3829 4003 4104 4218 4280 4370 4386 4592 INSTALL 0 INSTALL 0 INSTALL 0 13 243 384 866 1158 1525	
19.06.1990	7290	891082	93,05	2625	568
05.07.1990	7456	891082	95,44	2791	
13.07.1990	7636	891082	93,2	2791	
30.08.1990	8554	891082	95,2	3889	529

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DATE	PLANT	MODULE	R COND.	HOURS IN	FLUX
	HOURS	NO.	%	USE	ℓ/m².d
06.03.1989	0	881323	-	INSTALL 0	
01.06.1989	1047	881323	92	1047	
22.06.1989	1472	881323	90	1472	
28.07.1989	2190	881323	88,5	2190	
12.10.1989	3070	881323	67	3070	
13.11.1989	3686	881323	59	3686	
20.11.1989	3829	881323	55,6	3829	
30.11.1989	4003	881323	90,2	4003	
04.12.1989	4104	881323	56,1	4104	
11.12.1989	4218	881323	51,9	4218	
15.12.1989	4280	881323	48,7	4280	
22.12.1989	4370	881323	54	4370	
19.01.1990	4386	881323	51,8	4386	
29.01.1990	4592	881323	59,8	4592	
31.01.1990	4638	891092		INSTALL 0	
02.02.1990	4678	891092	93,6	40	
13.02.1990	4908	891092		270	
13.02.1990	4908	891091	-	INSTALL 0	
19.02.1990	5049	891091		141	
02.03.1990	5285	900034		INSTALLU	
12.03.1990	5531	900034	96,31	246	
26.03.1990	5823	900034	90,55	538	
11.04.1990	6190	900034	90,22	905	
22.04.1990	6303	900099		INSTALL U	770
19.06.1990	7290	900099	97,02	987	//9
12.07.1990	7400	900099	98,00	1103	
13.07.1990	/030	900099	97,25	1333	710
30.08.1990	8554	900099	97,3	2251	/19

#### POSITION NO. 18

DATE	PLANT	MODULE	R COND.	HOURS IN	FLUX
	HOURS	NO.	%	USE	<i>t</i> /m².d
06.03.1989	0	881319	•	INSTALL 0	
14.03.1989	125	881576	-	INSTALL 0	
01.06.1989	1047	881576	86	922	1
22.06.1989	1472	881576	86	1347	
28.07.1989	2190	881576	81,5	2065	
12.10.1989	3070	881576	56,5	2945	
13.10.1989	3092	881490	•	INSTALL 0	
13.11.1989	3686	881490	90	594	
20.11.1989	3829	881490	89,5	737	
30.11.1989	4003	881490	93,7	911	
04.12.1989	4104	881490	92	1012	
11.12.1989	4218	881490	89,4	1126	
15.12.1989	- 4280	881490	89,2	1188	
22.12.1989	4370	881490	90,4	1278	
19.01.1990	4386	881490	90,4	1294	
29.01.1990	4592	881490	94,3	1500	
02.02.1990	4678	881490	75,6	1586	
13.02.1990	4908	881490		1816	
19.02.1990	5049	881490		1957	
12.03.1990	5531	881490	85,01	2439	
26.03.1990	5823	881490	85,38	2731	
11.04.1990	6190	881490	85,62	3098	
19.06.1990	7290	881490	86,47	4198	625
05.07.1990	7456	881490	89,51	4364	
13.07.1990	7636	881490	84,06	4544	
30.08.1990	8554	881490	87,3	5462	610

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DATE	PLANT	MODULE	R COND.	HOURS IN	FLUX
	HOURS	NO.	%	USE	ℓ/m².d
06.03.1989		881206		INSTALLO	
29.04.1989	713	890024	-	INSTALLO	
01.06.1989	1047	890024	95	334	
22.06.1989	1472	890024	95	759	
28.07.1989	2190	890024	93	1477	
12.10.1989	3070	890024	87	2357	
14.10.1989	3112	881342	-	INSTALL 0	
13.11.1989	3686	881342	89,7	574	
20.11.1989	3829	881342	91,7	717	
30.11.1989	4003	881342	93,8	891	
04.12.1989	4104	881342	92,2	992	
11.12.1989	4218	881342	90,2	1106	
22 12 1009	4200	991342	09,4	1258	
10 01 1000	4396	881342	91,2	1230	
29.01.1990	4592	881342	94.2	1480	
02.02.1990	4678	891080	-	INSTALL 0	
02.02.1990	4678	891080	95.1	0	
13.02.1990	4908	891080		230	
19.02.1990	5049	891080		471	
12.03.1990	5531	891080	94,08	853	
26.03.1990	5823	891080	94,84	1145	
11.04.1990	6190	891080	94,09	1512	
19.06.1990	7290	891080	92,42	2612	578
05.07.1990	7456	891080	95,78	2778	
13.07.1990	7636	891080	93,5	2958	000
30.08.1990	8554	891080	93,6	3876	608

#### POSITION NO. 20

DATE	PLANT	MODULE	R COND.	HOURS IN	FLUX
	HOURS	NO.	%	USE	北/m <sup>2</sup> .d
06.03.1989 01.06.1989 22.06.1989 28.07.1989 12.10.1989 13.11.1989 20.11.1989 30.11.1989 04.12.1989 15.12.1989 15.12.1989 15.12.1989 22.12.1989 19.01.1990 31.01.1990 02.02.1990 13.02.1990 12.03.1990 12.03.1990 26.03.1990	HOURS 0 1047 1472 2190 3070 3686 3829 4003 4104 4218 4280 4370 4386 4592 4638 4578 4678 4678 4678 4678 4592 5531 5823 5100	NO. 881297 891030	% 88 84,5 82,5 61,5 47,9 90,9 63,5 45,3 41,9 40,8 43,9 40,8 43,9 45,6 51,1 - - 98,9 94,06 94,43 94,06	USE INSTALL 0 1047 1472 2190 3070 3686 3829 4003 4104 4218 4280 4370 4386 4392 INSTALL 0 INSTALL 0 INSTALL 0 0 230 371 853 1145 1512	<i>t</i> /m².d
19.06.1990	7290	891030	93,19	2612	574
05.07.1990	7456	891030	95,55	2778	
13.07.1990	7636	891030	95,60	2958	
30.08.1990	8554	891030	j 94,10	3876	672

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APPENDIX IV CALCIUM SULPHATE SEED PARTICLE SIZE CHARACTERISTICS

## APPENDIX IV

# MLT\_PLANT : PARTICLE SIZE OF SEED d50 MICRONS

Date	Brine Reject	Cyclone Overflow	Cyclone Underflow	Feed	Gypsum
19.09.89	30	12 .	29	28	
20.09.89	37			44	
05.10.89	41	41	40		
11.10.89	39	28	42		
19.10.89	45	15	44		
25.10.89	42	20	44		
02.11.89	43	35	43		
08.11.89	42	23	43		
15.11.89	41	28	42		
22.11.89	40				
29.11.89	30	13	44		
07.12.89	37	25	37		
13.12.89	30				
19.12.89	40				
01.02.90	40	11	40		
08.02.90	45		50		
15.02.90	50	10	70		
22.02.90	40	12	45		
01.03.90	42	14	42		
07.03.90	32	11	40		
14.03.90	30	11	35	30	
15.03.90					40

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Date	Brine Reject	Cyclone Overflow	Cyclone Underflow	Feed	Gypsum
19.09.89	68	35		64	75
05.10.89	66	37	67		
11.10.89	37	35	51		
19.10.89			25		
25.10.89	44	38	43		
02.11.89	49	37	48		
15.11.89	60	50	60		
22.11.89	65				
29.11.89	55	55	60		
07.12.89	60	70	62		
13.12.89	65				
01.02.90	60	50	60		
08.02.90	84	12	80		
15.02.90	71	11	90		
07.03.90	55	10	50		
14.03.90	50	50	50		

# SPARRO PLANT : PARTICLE SIZE OF SEED d<sub>50</sub> MICRONS



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12 D. 44



0,1 0,2 0,5 1 2 5 10 20 50 100 200 500 1 000

113

APPENDIX V MICROSCOPE PHOTOGRAPHS OF CELLULOSE ACETATE MEMBRANE THAT FAILED IN SPARRO PLANT

APPENDIX V MICROSCOPE PHOTOGRAPHS OF CELLULOSE ACETATE MEMBRANE

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## APPENDIX V

## MICROSCOPE PHOTOGRAPHS OF CELLULOSE ACETATE MEMBRANE THAT FAILED IN SPARRO PLANT





# MICROSCOPE PHOTOGRAPHS OF CA MEMBRANE FROM MODULE EX BANK 4 (X 200) AFTER 285 HOURS OPERATION

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APPENDIX VI MICROSCOPE PHOTOGRAPHS OF CALCIUM SULPHATE SEED CRYSTALS FROM MLT PLANT AND COMMERCIAL GYPSUM

APPENDIX VI

MICROSCOPE PHOTOGRAPHS OF CALCIUM SULPHATE SEED CRYSTALS

CALCIUM SULPHATE SEED CRYSTALS EX MLT PLANT (X 200)



COMMERCIAL GRADE GYPSUM USED AS SEED (X 200)

APPENDIX VII ANALYSIS OF COMRO TUBULAR CA MEMBRANES (1989)

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#### APPENDIX VIII

#### ANALYSIS OF COMRO TUBULAR CA MEMBRANES

#### INTRODUCTION

No chemical analyses were done, to determine the extent of possible hydrolysis of the COMRO membranes received from MEMBRATEK. The titration techniques described in the literature for determining the hydroxyl or acetyl content of a cellulose acetate polymer are regarded as insufficiently sensitive and accurate at the levels at which chemical modification (due to hydrolysis) of the membrane surface would have a serious effect on membrane performance. For the same reason IR spectral analysis of the membrane material was not considered worthwhile apart from the fact that the Institute itself does not possess the necessary analytical equipment to perform any such quantitative tests.

The membrane samples were compared by means of optical microscopy and dye staining. Samples were also preserved for comparison with membranes to be received at a later date.

#### **REFLECTED-LIGHT DIC MICROSCOPY - MEMBRANE SURFACE DETERIORATION**

This optical analytical technique is designed especially for the investigation of smooth-surfaced objects. The method used highlights surface defects (deformations) and, for that reason, is ideally suited to investigation of the topography of a membrane surface; scanning electron microscopy is not suitable.

Sections were cut from the membranes received. The membrane films were not removed from the support fabric, and the samples were mounted onto glass slides by means of double-sided masking tape. The membranes were not rinsed clean during preparation of the samples, and great care was taken not to damage the sample surfaces. Care was also taken to note the axial cross-flow direction, and any force applied to the membrane surface during mounting of the sample, was applied perpendicular to the direction of flow.

All the samples were gold-shadowed before microscopic analysis. Photographic records were made of each of the different samples analysed. The photographs have a final magnification of 1 000x.

In order to have some sort of reference, samples of CA membranes that originated from two other process applications were also obtained. As a control, fresh, unused CA membranes were included in the study.

The following membranes were analysed:

1. Set A: Unused membranes which were taken directly from production storage.

- 2. Set B: A set of membranes (taken from the inlet, centre and outlet sections of the module) was taken from a module in an RO pilot plant operating on filtered final effluent from an extended aeration sewage plant as feed. (These membranes have been subjected to sponge ball cleaning on occasions).
- 3. Set C: These membranes originated from an RO application in which small soft crystals of an organic acid are formed during concentration of the feed. No sponge ball cleaning was performed during the period of operation of these membranes.
- 4. Set 1: The COMRO membranes received as Sample No. 1 (881324).
- 5. Set 2: The COMRO membranes received as Sample No. 2 (881320).

#### SURFACE DEPOSITS - OPTICAL AND SEM ANALYSIS

Our Department of Physics has an EDAX facility coupled to their SEM, which would make elemental analysis of the surface deposits possible in principle. Non-adhering crystals were found to be present on the surfaces of the membrane samples investigated, but no evidence was found of fixed deposits or scale.

As an after thought, membrane samples could be rinsed clean with distilled water, the water evaporated, and the collected debris mounted on a carbon stub for elemental analysis. This would give an indication of the collective identity of substances of inorganic nature present within a membrane tube.

#### SURFACE STAINING - SIGNS OF MEMBRANE MODIFICATION

All the COMRO samples received were subjected to dye staining with an Identification Stain (Du Pont). No photographic records were made, as colour comparison of different membranes over a period of time would virtually be impossible, according to the photographic experts. However, it can be done if all the photographs were to be taken in a single session and the film processed in a single batch.

#### RESULTS

#### Microscopic Study

Figure 1 shows the surfaces of unused membranes. There is a marked difference between these membranes and those shown in Figures 4 and 5 which represent COMRO samples No. 1 and 2, respectively.

The streak lines on the membrane surface, caused by scraping of the sponge balls against the membrane, can be seen clearly in Figure 2. No other marks, except the scrape lines and dried bio-film, were present on any of the samples investigated.

Some collision of soft crystals with the membrane surface is evident from the three records shown in Figure 3. These membranes have apparently been operated for several months in a process in which crystals of an organic acid formed during concentration of the feed.

In comparison with Figure 3, Figures 4 and 5 show considerable surface deformation due to impingement of crystal. The direction of fluid-flow, which incidentally is the same on all the photographs, is clearly illustrated as along the length of the photograph.

It appears possible that continuous sustained exposure of the membrane surface to particle bombardment may eventually erode the top layer of the membrane and cause a drop in performance. It appears at present that the deformation which has taken place was purely plastic. However, although no evidence of surface puncturing could be found, the possibility that this has occurred cannot be ruled out completely, as some of the scouring marks noticeable are up to 0,5 µm wide.

#### Dye Staining

The membranes in both the COMRO sample Sets Nos. 1 and 2 (inlet, centre and outlet samples) stained the same light-yellow tint as did the MEMBRATEK set of unused control membranes.

Furthermore, the COMRO membranes all had radial indentations which corresponded to the parting line between the plastic pressure support disks of the tubular module. In addition to the light-yellow tint, the COMRO membranes also showed slight red discolouration along some of these indentation lines. An unused control membrane sample, which had been exposed to a 10 per cent NaOH solution for 5 minutes, stained a shade of green when it was subjected to a similar dye test. It is uncertain whether the red stain can be ascribed to a chemical or physical alteration of the membrane surface/material.

From fluid dynamic considerations it is however to be expected that concentration build-up in the indented areas would be higher than on the ridges between the indents. The likely occurrence of hydrolysis would, for the same reason, be higher in these areas.

Samples of the sets of membranes already received will be preserved and used for comparison in staining experiments to be conducted on future membranes to establish qualitatively whether the membrane actually deteriorates along the indentation lines.

## **SUGGESTION**

The possibility that a protective coating might reduce or prevent erosion of the membrane surface could be considered.

Dr E P Jacobs Institute for Polymer Science University of Stellenbosch

24 July 1989 Report COMRO1

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# Figure 1 MICROGRAPHS (1000X MAGNIFICATION) OF THE SURFACE OF TUBULAR CA MEMBRANES - FRESH, UNUSED





INLET



CENTRE



Figure 1 MICROGRAPHS (1000X MAGNIFICATION) OF THE SURFACE OF TUBULAR CA MEMBRANES - FRESH, UNUSED

OUTLET

# Figure 2 MICROGRAPHS (1000X MAGNIFICATION) OF THE SURFACE OF TUBULAR CA MEMBRANES - TREATED SEWAGE





INLET



CENTRE

## CONTINUED

# Figure 2 MICROGRAPHS (1000X MAGNIFICATION) OF THE SURFACE OF TUBULAR CA MEMBRANES - TREATED SEWAGE



## OUTLET

# Figure 3 MICROGRAPHS (1000X MAGNIFICATION) OF THE SURFACE OF TUBULAR CA MEMBRANES - ORGANIC ACID CRYSTALS PRESENT



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INLET

CENTRE

# CONTINUED

# Figure 3 MICROGRAPHS (1000X MAGNIFICATION) OF THE SURFACE OF TUBULAR CA MEMBRANES - ORGANIC ACID CRYSTALS PRESENT



OUTLET

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# Figure 4 MICROGRAPHS (1000X MAGNIFICATION) OF THE SURFACE OF TUBULAR CA MEMBRANES - SEEDED SLURRY - COMRO MEMBRANES





CENTRE

## CONTINUED

# Figure 4 MICROGRAPHS (1000X MAGNIFICATION) OF THE SURFACE OF TUBULAR CA MEMBRANES - SEEDED SLURRY - COMRO MEMBRANES



## OUTLET

Figure 5 MICROGRAPHS (1000X MAGNIFICATION) OF THE SURFACE OF TUBULAR CA MEMBRANES - SEEDED SLURRY - COMRO MEMBRANES





INLET



CENTRE

# CONTINUED

# Figure 5 MICROGRAPHS (1000X MAGNIFICATION) OF THE SURFACE OF TUBULAR CA MEMBRANES - SEEDED SLURRY - COMRO MEMBRANES



## OUTLET
# ANALYSIS OF MEMBRANE SAMPLES SECOND MODULE SAMPLE SET

# INTRODUCTION

Two sets of membrane samples were received from Membratek for analysis, one set marked Module 3 (881295) and the other Module 4 (881502); each set of membranes taken from the inlet, centre and outlet of each module.

# **OPTICAL MICROSCOPY** - Differential Interference Contrast (DIC)

#### Sample Preparation

Samples were prepared for optical microscopy by cutting 6 cm-lengths from the various tubes. These samples were mounted on glass slides, and in order to get some indication of possible foulant deposits, the surfaces of the membrane samples were never touched by hand during sample preparation.

The samples were allowed to dry out naturally and were sputter-coated with gold before being analysed.

#### Observations

There were marked differences between the appearance of membranes from Modules 3 and 4 and those of Modules 1 and 2 received previously. Radial bands of up to 14 mm, and dull in contrast to the remainder of the membrane films which were still shiny in appearance, extended at intervals across the length of the samples analysed. Initially this was observed only after sputter-coating with gold. However, when once observed, it was also recognized on other samples once the surfaces of the membranes were wiped clean of any loose surface deposits or debris.

DIC micrographs were taken of sections of the various samples received (1000x magnification). As in the case of Modules 1 and 2 earlier investigated (Report COMRO1), erosion marks, caused by particle impingement, were again present as can be seen from Figure 1 (Module 3: Inlet, centre and outlet) and Figure 2 (Module 4: Inlet, centre and outlet).

Deposits were present in a greater or lesser extent on all of the samples analysed. (Module 3 seems to be less affected than Module 4). Cases were observed where deposits seem to have nucleated from scour marks left by larger-sized particle impingement on the membrane surface. (Figure 3 is an example of this occurring in Module 3: Inlet and centre sections). Figure 4 shows evidence of some scale deposit (Module 3: Centre and outlet).

Except for brief examination during EDAX analysis, the membranes were not investigated by means of SEM. During optical microscopy evaluation of the various samples, no obvious evidence was found of gross structural damage to the membrane body.

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However, a strange phenomenon was noticed during the analysis of samples from Module 4 membranes. Previously, the marks on the membrane surfaces were all essentially unidirectional, and clearly in the axial direction of fluid flow. In Module 4 (see Figure 5), the membrane surface carried impressions which were clearly not unidirectional. A possible explanation for these randomly orientated marks is that they were formed by crystals of some sort, which had been forced into the membrane surface under the action of hydrostatic pressure. However, no evidence of the presence of long-shaped crystals could be found anywhere during microscopic analysis.

# <u>STAINING</u>

Samples from the various membrane sets were rinsed clean with a 5 per cent acetic acid solution and rinsed thoroughly before being stained with identification stains obtained from Du Pont.

In comparison to membranes from the earlier received modules, membranes from Modules 3 and 4 differed in the way in which they stained under the action of the dye. This was regarded as some indication that the chemistry of the membrane material may have been affected.

#### FOURIER TRANSFORM INFRARED (FTIR)

To gain some proof for the above observation, membrane samples were taken for FTIR analysis. The samples were analysed using the ATR facility on the instrument as the membrane films abstracted from the support fabric were not clear. Unfortunately the spectra obtained during this part of the exercise were of little value in the end due to low emission levels.

However, during experiments to dissolve membrane films taken from Modules 3 and 4 and membrane films taken from freshly produced CA membranes (Membratek supplied) and virgin CA 398-10 cellulose di-acetate powder (IPS stock), differences in solubility behaviour was noticed.

Virgin membrane films and powder dissolved in acetone and swelled in chloroform. However, Module 3 and 4 samples only swelled in acetone, whereas chloroform left the films unaffected.

This was thought to be in further evidence that the chemistry of the membranes had been modified, although it could not be quantified due to the unavailability of the FTIR equipment.

#### EDAX ANALYSIS OF DEPOSITS ON MEMBRANE SURFACE

Specimen membranes were carbon-coated for elemental analysis of surface deposits by EDAX.

A representative elemental analysis of the deposits is shown in an EDAX printout (Figure 6). According to this analysis there is little presence of CaSO<sub>4</sub> in the deposit. Deposits appear mainly to consist of aluminium and silicon (probably aluminium silicates), and to a lesser extent iron and calcium (sulphates). (In retrospect, the tiny (< 0,1  $\mu$ m) crystals that were seen during polarized light microscopy is therefore most probably of quartz origin).

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# **CONCLUSION**

Without the benefit of comparing the analysis given to that of a module that has accumulated the same number of operating hours, and which is still in good performance, the following can be said:

- 1. The surfaces of the membranes analysed appear to be eroded to a greater extent than those of Modules 1 and 2 (report COMRO1).
- 2. Surface deposits, consisting mainly of aluminium-silicates, were observed, especially in the case of Module 4.
- 3. According to solvent solubility differences, the COMRO samples are believed to be chemically different from the virgin membrane samples and powder analysed. (However, the latter is very much dependant on whether the same source and grade of material was used as reference material).
- 4. Based on the samples analysed from Modules 3 and 4, no reason, other than the possibility of hydrolysis, can be offered for the lower performance of the membrane specimens received.

# **SUGGESTIONS**

- The Institute will approach Membratek for 500 mm-sections of membranes from Modules 3 and/or 4. The membranes will be coated with supplemental polymers to establish whether it would be possible to restore the performance of the membranes. If positive results are obtained, arrangements can be made for module evaluation and dissemination of the results of the study.
- The IPS can make tubular membrane test cells (400 500 mm long) available to COMRO, on either a hire or purchase basis, to allow close observation of single membrane performance. (Such cells can be piped into the end of a module train). This offers the advantage that single membranes can be removed for inspection at the first sign of performance deterioration. This might allow some insight into possible cause of membrane failure.

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Figure 1 SURFACE EROSION PRESENT IN MODULE 3 (Inlet, centre and outlet)

CENTRE

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OUTLET



Figure 2 SURFACE EROSION PRESENT IN MODULE 4 (Inlet, centre and outlet)

CENTRE



OUTLET

Figure 3 <u>ALUMINIUM/SILICATE-RICH DEPOSITS NUCLEATING FROM SURFACE EROSION</u> MARKS (Module 3: Inlet, centre)





CENTRE

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Figure 4 SURFACE DEPOSITS FOUND PRESENT IN MODULE 3 AND 4 (Module 3: Inlet, centre)

CENTRE



Figure 5 RANDOM DIRECTION IMPRESSIONS FOUND PREDOMINANTLY IN MODULE 4 (Module 4: Centre, Centre, Outlet)

CENTRE



OUTLET

# Figure 6 EDAX SPECTRA AND EXPLANATION OF ELEMENTAL ANALYSIS REPRESENTATIVE OF FOULANT LAYER FOUND IN MODULE 4



