IPC

The KNEW Process

An Assessment of the Concept

Industrial & Petrochemical Consultants (Pty) Ltd

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Executive Summary

The proposal to develop a process to treat acid mine water by ion exchange removal of the heavy metals, followed by ion exchange removal of calcium, magnesium and sulphate, and production of, primarily, a potassium nitrate product was reviewed. It is the proposer's intention to construct a pilot plant to demonstrate this process, and the review showed this to be appropriate, as there remain a few areas of uncertainty that should be resolvable by piloting.

It is concluded that it is not possible at this stage of the development to express a firm opinion about the economics of the process, primarily because the capital cost of any full scale plant is presently a little too uncertain. On what has been presented, we are confident that the pilot plant work should clear up any uncertainties to a level sufficient to enable the full-scale plant to be designed and costed with confidence. It is, however, true to say that if the pilot plant operates close to specification, then it seems unlikely that the capital cost of a full scale plant will inflate greatly over present estimates, which would indicate a profitable process.

Introduction

Trailblazer Technologies (Pty) Ltd has requested IPC to review its proposal to ensure that it is as realistic as possible at this stage of its development. Material was provided to IPC to enable this review to take place as follows:

1. A presentation entitled “Acid Mines Water – Exxaro presentation 10-11”
2. An Excel spreadsheet “AMD mat bal & costing 1.4Mpd Namakwa Sands 11-11”
3. A Powerpoint presentation of a single slide “METCON Evaporation pic”
4. An Excel spreadsheet “Sunday’s River material balance and costing 10-10”
5. A single page document entitled “Trailblazer Technologies KNEW process.”
6. Ten Piping and Instrumentation Diagrams (P&ID’s).
7. Specifications for ion exchange resins
8. An analysis for sodium and chloride of potassium nitrate produced by the process

IPC’s capabilities are outlined in the Appendix to this report. Much of our process review work has been at a much later stage in the development of a process. Our review work has included checking of mass and energy balances, PFD’s and P&ID’s, HAZOP and HAZAN reviews, and operability and maintainability reviews.

In addressing the present request, we are very cognisant of the limited scope of the information provided, indicative of a process more at the conceptual stage than at the prefeasibility stage. We note that it is intended to construct a pilot plant to verify the assumptions that it was necessary to make in deriving the limited information on which the concept is based. The P&ID’s, however, gave evidence that the concept had been reasonably completely thought through. In what follows, therefore, it should be noted that we have attempted to identify the assumptions, and then to assess whether, in our expert opinion, they are reasonable or whether there are significant foreseeable risks associated with the assumptions, such that more work is necessary before a pilot study should be undertaken.

We have reviewed the costing information provided, and can report that it is perfectly reasonable, and that provided the technical aspects of the project can be proven by pilot plant tests, then it seems entirely possible that an economic process could result.

Review

The process comprises an ion exchange system to:

1. Remove a heavy metal fraction from the feed by ion exchange
2. Separate the cleaned feed into cation-rich and anion-rich fractions by ion exchange
3. The cation exchange resin is regenerated with nitric acid, and the cation-rich fraction is treated with carbonate to remove calcium and magnesium. The liquor is then treated with a potassium-rich reagent and concentrated in a direct-contact heat exchanger. Sodium chloride crystals precipitate from the hot concentrated liquor and potassium nitrate from the supernatant after sodium chloride precipitation.
4. The anion exchange resin is regenerated with ammonia solution, and ammonium sulphate is recovered from the liquor by treatment with methanol, the methanol being recovered by distillation and recycled.

Figure 1, taken from Reference 1 in the list in the Introduction, shows the ion exchange section.

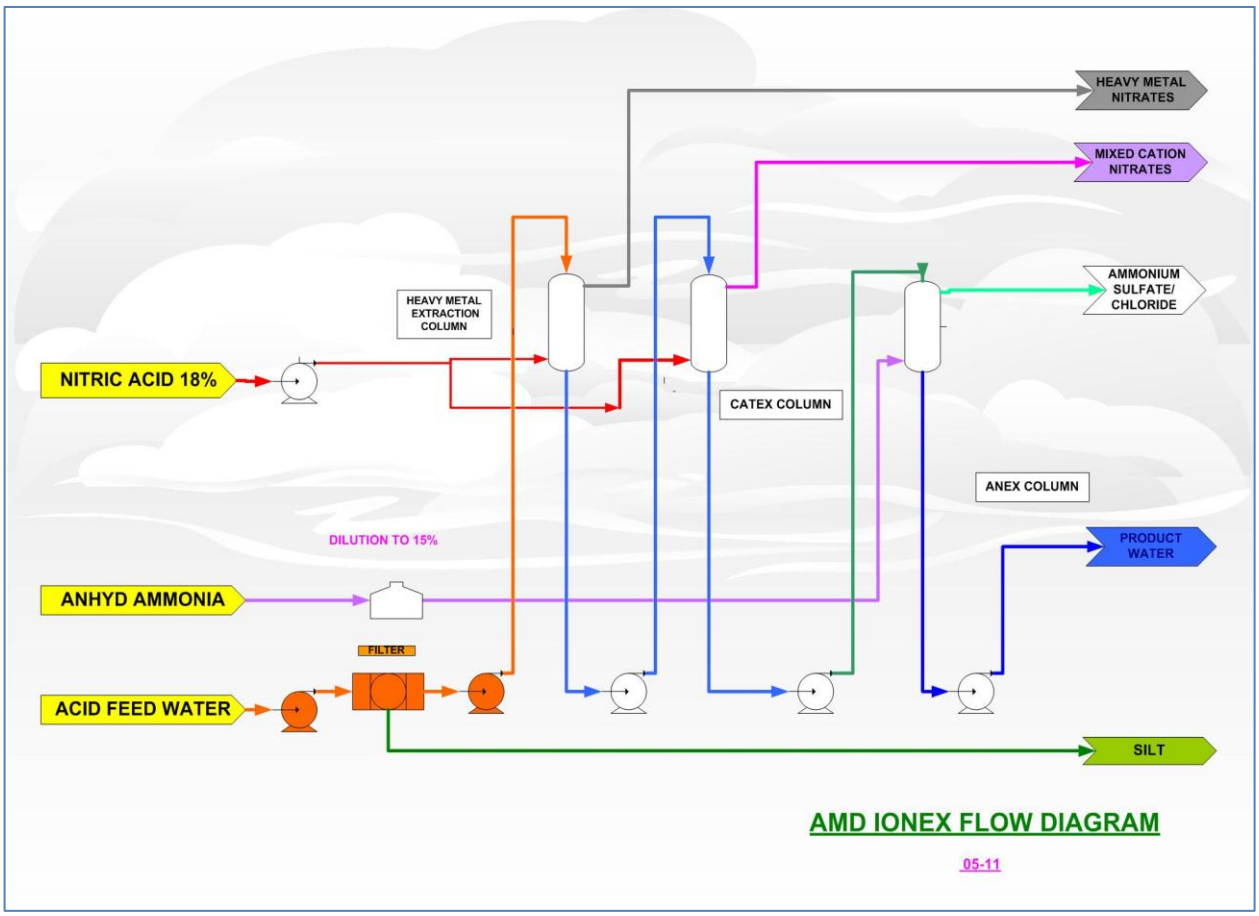

Figure 1 The ion exchange system

The acid feed is clarified before passing to a 'Heavy Metal Extraction Column'. The heavy metals, iron and often manganese in the usual acid mine drainage, and possibly also uranium and its decay

products (thorium, radium, bismuth, thallium, protoactinium and lead being the longer-lived species) are held on the ion exchanger while lighter metals Ca, Mg and Na pass on to the next stage. Note that only one column is shown; in the P&ID's, three are used, one of which would be being loaded with heavy metals; one of which would be scavenging the tail from the first; and the third would be being regenerated. An assumption is that the **ion exchanger would be successfully regenerated with moderately strong nitric acid** [1]. There is a technical risk in this – nitric acid is a moderately strong oxidizer, and not all ion exchange resins are resistant to oxidation, particularly when repeatedly recycled. In the early days of the South African uranium industry, anion exchange resins were used to absorb uranium selectively, and were then regenerated with nitric acid. The resin life was significantly below expectation because of oxidative phenomena. However, resin manufacturers have indicated that a 5-year life seems reasonable in this service, so a 20% per annum depreciation allowance on resin inventory is rational. Further assumptions are that **the heavy metals will be recoverable from the nitric acid eluate** and that **it will be possible either to dump or otherwise dispose of the heavy metals**. If the stream does indeed contain significant quantities of radioactive species, then dumping would probably not be possible. More work is necessary on this aspect.

The water now stripped of its heavy metal content would now pass to a second ion exchanger, the CATEX column in Figure 1, to remove Ca and Mg preferentially. A step that is missing is **pH adjustment after the Heavy Metal Extraction Column**. The effluent from the Heavy Metal Column will be more acid than the feed because in the column,

$$Me^{++} + RH_2 \rightarrow RMe + 2H^{+} \quad \text{---------- (1)}$$

Some alkali will therefore be needed to neutralize the extra acid.

Equation (1) above will operate in the CATEX columns also, and the concentration of Ca and Mg in the feed will be relatively high, so their exchange will increase the pH again. This interferes with the exchange, because the hydrogen ions compete with the metal ions, and **the pH may need to be readjusted between columns**. It is for this reason that mixed resins are often used in water softening. Sodium will probably not be fully picked up in the CATEX column, because it is more weakly absorbed than Ca or Mg, so **the final product water will contain some sodium**.

The effluent from CATEX passes to ANEX, the anion exchange column to remove sulphate preferentially:

$$SO_4^{++} + R(OH)_2 \rightarrow RSO_4 + 2OH^{-} \quad \text{---------- (2)}$$

Equation (2) above will operate in the ANEX columns also, and the concentration of sulphate in the feed will be relatively high, so its exchange will reduce the pH. This interferes with the exchange, because the hydroxyl ions compete with the sulphate ions, and **the pH may need to be readjusted between columns**.

[1] Items in **bold** refer to assumptions or uncertainties, the impacts of which are discussed in the next section

Turning now to the elution cycles in the ion exchange circuit of Figure 1, the heavy metals and the Ca and Mg should all be satisfactorily and rapidly eluted by nitric acid. The possibility of reduced resin life due to oxidation by nitric acid has already been noted. In the ANEX section, elution with ammonium hydroxide should also be satisfactory and rapid.

Before leaving consideration of the ion-exchange section of the plant, some remarks about materials of construction seem warranted. We were provided with no information on this aspect. The use of nitric acid to elute both the heavy metal column and the CATEX column makes the use of stainless steel almost mandatory. However, **the choice is critically dependent on the chloride concentration in the feed stream**, as small quantities of chlorides can cause stress corrosion cracking in the lower grades (the 18-8) stainless steels. An alternative is to employ rubber-lined mild steel, which tends to get rather costly on complex shapes such as distributors in columns. Many chemical grade plastics are perfectly resistant and can be employed in this duty. However, it needs to borne in mind that this is the design of a pilot plant, not a full-scale plant, and it is not as critical to specify materials to the same degree as in a full-scale plant.

Figure 2 reproduces the proposed workup of the CATEX eluate. The mixed nitrates in the eluate, primarily calcium and magnesium nitrates, are mixed with sodium carbonate ('soda ash') which precipitates the calcium and magnesium as insoluble carbonates:

$$Ca^{++}\ (Mg^{++}) + CO_3^{--} \rightarrow Ca(Mg)CO_3 \quad \text{---------- (3)}$$

It should be noted that this step is described as 'optional'. It is unclear what would happen to the calcium and magnesium in the potassium recovery step if this step were omitted. However, we note that many acid mine waters are in fact low in calcium and magnesium (provided that have not been treated with lime or dolomite) so under these conditions omission of this step is reasonable. It should also be noted that it is proposed that a stream described as "ANEX CHLORIDE M/L" is added, which is a sodium chloride-rich mother liquor produced during ammonium sulphate production. The naming of this stream is therefore misleading. The mixed carbonate precipitate is then centrifuged to recover the mother liquor. **It may be necessary to add a wash step to recover all the potassium from the centrifuge product**.

The liquor is then mixed with potassium chloride, further soda ash and recycled potassium-rich mother liquor. An agitator is shown, but if no precipitation takes place (particularly if calcium and magnesium are largely removed by the prior optional step) **then this could be replaced by an in-line mixer.**

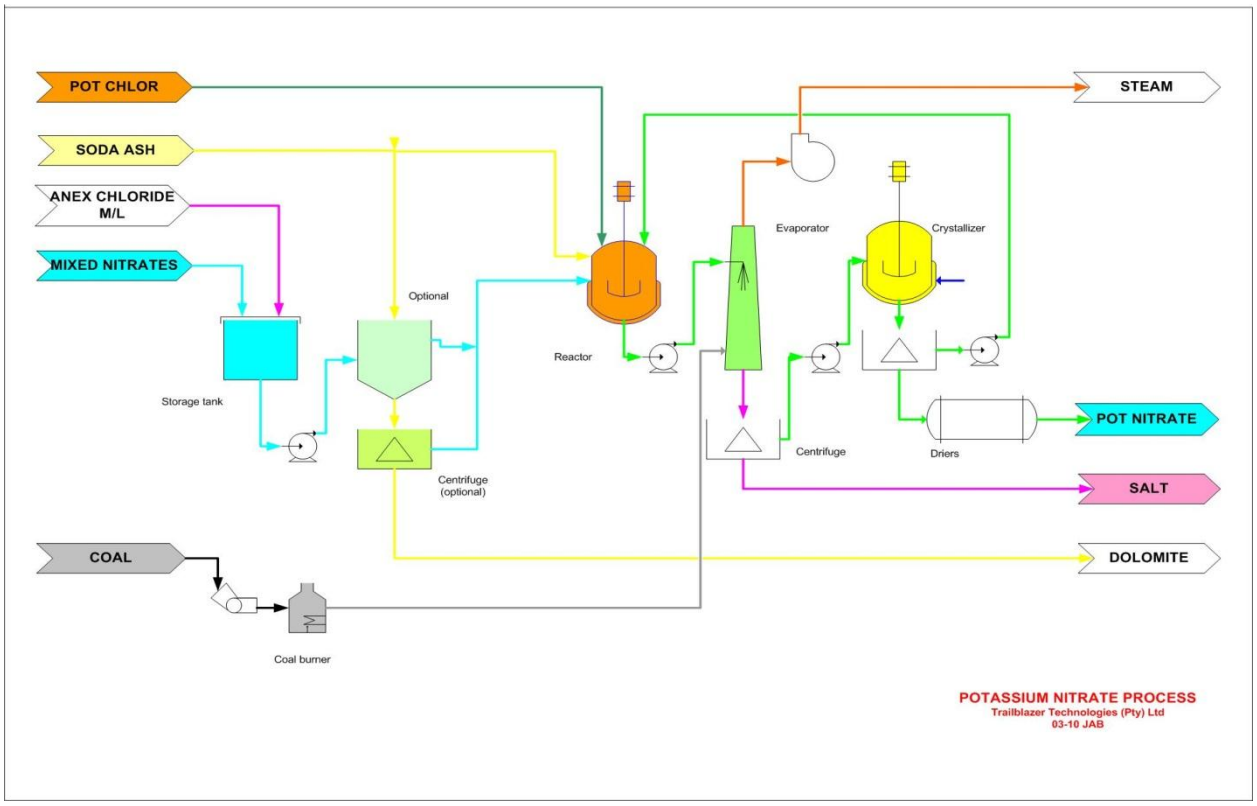

Figure 2 Flow diagram for proposed potassium nitrate production

The mixture passes to a direct-contact evaporator in which the concentrations of sodium and potassium are increased and water removed by heat. The removed water is shown as “steam” but being at atmospheric pressure and comprising water vapour mixed with the products of combustion, there is no value in this stream. It should rather be shown as “to stack” or some such.

The concentrated liquor from the evaporator passes directly to a centrifuge. The sodium chloride has a lower solubility at high temperatures than potassium nitrate, as shown in Figure 3, so the sodium chloride will crystallize preferentially. This process has previously been applied successfully in the Chilean nitrate industry to separate sodium and potassium. Examination of Figure 3 shows that sodium nitrate is almost as soluble as potassium nitrate, and potassium chloride almost as insoluble as sodium chloride, at high temperatures. Thus the ‘sodium chloride’ crystallizing out from the concentrated liquor will contain some potassium chloride, and the mother liquor would contain a significant concentration of sodium in the nominal “potassium nitrate” stream.

The final step in this separation is the cooling of the liquor after centrifuge separation of the solid sodium chloride stream, when the potassium nitrate should preferentially crystallize. Figure 3 shows that this would probably be effective below about 65^{o}C, when any sodium would stay in solution as chloride and nitrate salts while potassium nitrate crystallized. **It seems to be untested**, but the risk of failure is low.

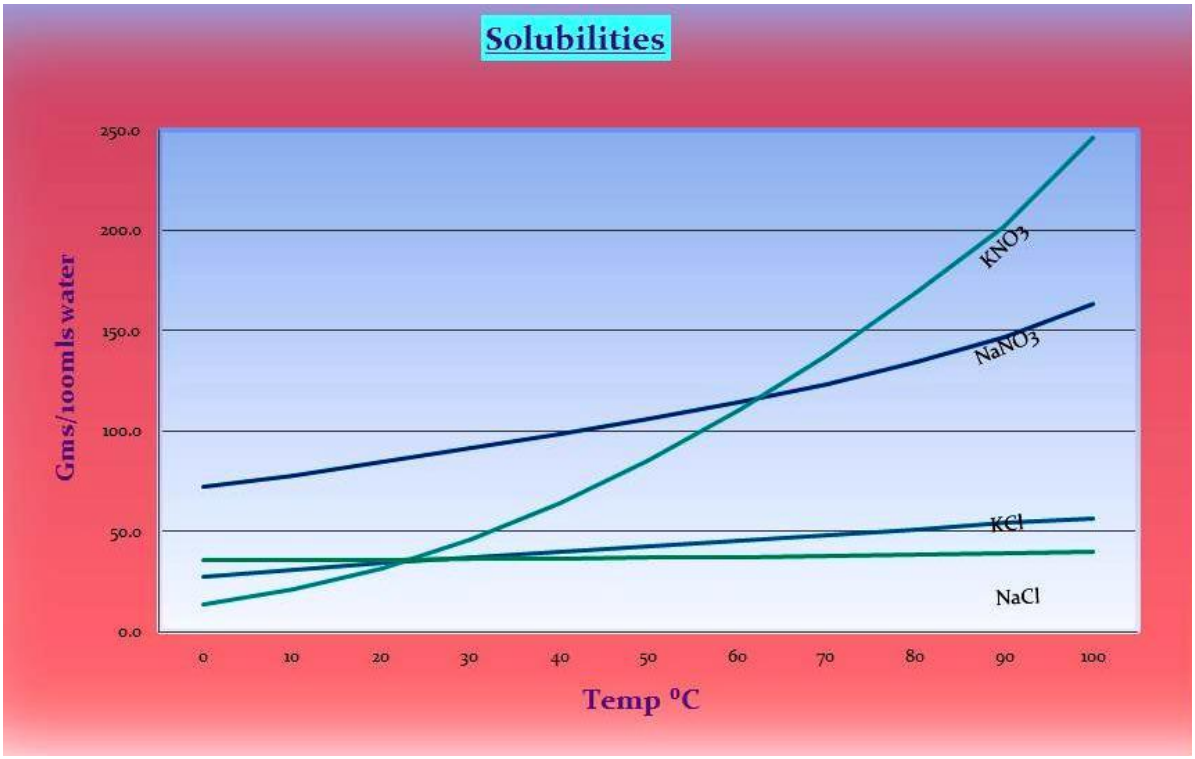

Figure 3 Solubilities of sodium and potassium nitrates and chlorides

We were shown an analysis of potassium nitrate produced in the laboratory by this process, and indeed the product contained less than 1% sodium and chloride, so a reasonably clean separation is certainly possible. There may be merit in running the process through a computer program such as Aspen Plus, which can model systems such as this quite accurately, so reducing the risk to very tolerable levels without great expense.

The sodium chloride contaminated with potassium chloride is worked up in another section (see below).

Finally ammonium sulphate and pure sodium chloride are produced in the flowsheet of Figure 4. The ammonium sulphate stream from the elution of the anion exchange columns is mixed with methanol, which precipitates ammonium sulphate. **We have been assured that this step has been tested successfully in the laboratory**, but we have seen no independent results. However, a pilot plant test should allow the flows and compositions to be quantified, and ammonium sulphate is only a moderate contributor to the revenues, so there is a little leeway in this step.

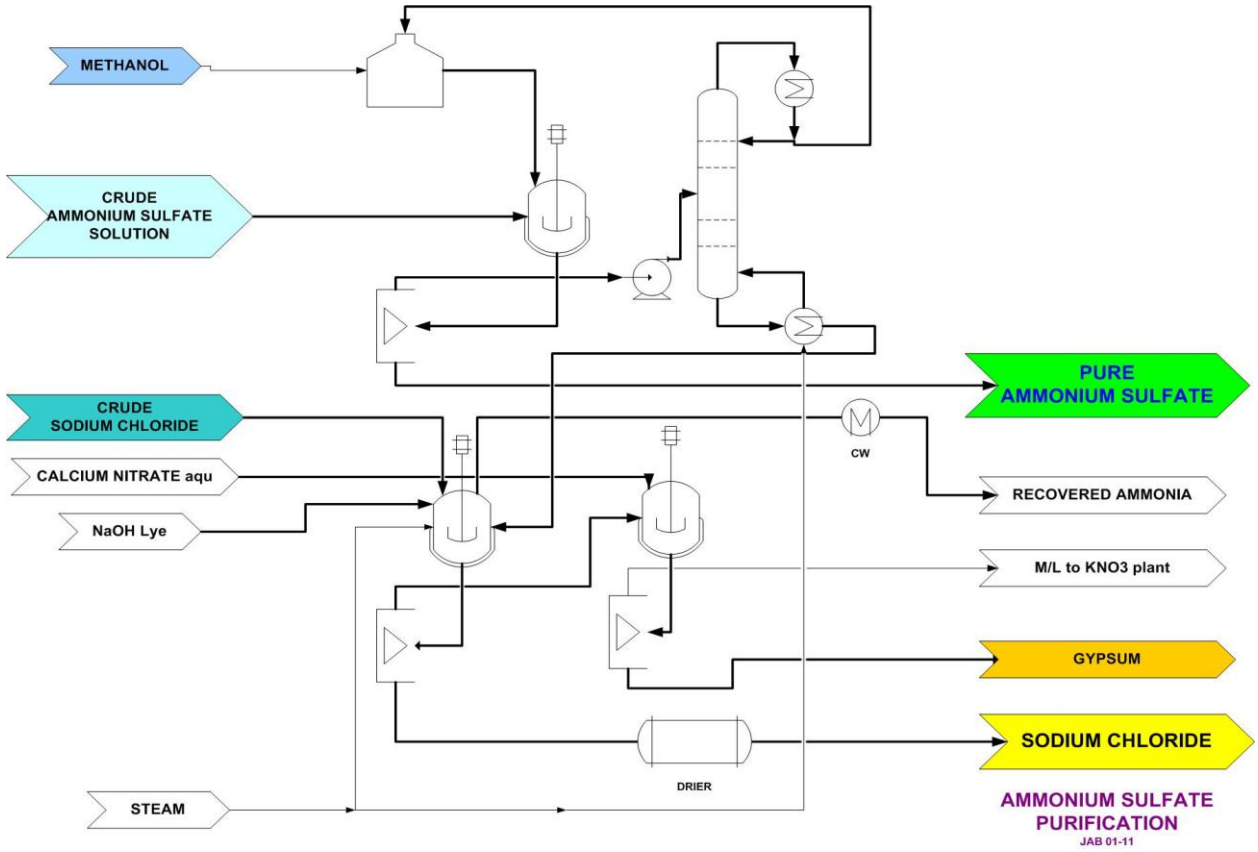

Figure 4 Production of pure ammonium sulphate and sodium chloride

The ammonium sulphate crystals are recovered by centrifuging and the supernatant liquid is sent to a methanol stripping column where the methanol is recovered and recycled. There is presumably some washing of the crystals to remove residual liquid **but this is not shown**. The aqueous phase from the bottom of the stripping column is added to the sodium chloride section.

Assessment of Identified Assumptions

The assumptions shown in the previous section are now considered in order, and their possible impact assessed:

1. **the ion exchanger would be successfully regenerated with moderately strong nitric acid**. The risk of degradation during regeneration is real, but it should be measured during the pilot plant trial so that the cost implications can be quantified.
2. **it will be possible either to dump or otherwise dispose of the heavy metals.** At the pilot plant stage this is not a critical issue, but will need to be addressed at the full scale
3. **A step that is missing is pH adjustment after the Heavy Metal Extraction Column.** This detail has only small cost implications.
4. **the final product water will contain some sodium**. This should not affect the overall process economics badly, and the impact will depend to a large extent on the sodium content of the feed.

5. **the pH may need to be readjusted between columns**. This detail has only small cost implications.
6. **the choice is critically dependent on the chloride concentration in the feed stream.** As noted in the body of the text, this is not critical at the pilot plant stage, but needs to be taken into account in the design of any full scale plant.
7. **It seems to be untested** While this is true, again it underlies the whole purpose of constructing a pilot plant and, on the basis of the data presented in Figure 3, we believe the chances of this being a source of risk are low.
8. **We have been assured that this step has been tested successfully in the laboratory.** It is not obvious why there will be selective crystallization of the ammonium sulphate in this case, although we have been assured that it is successful. However, the pilot plant will test this aspect.
9. **but this is not shown.** It is not certain what the need for a wash will be.

Conclusion

This review has identified a number of uncertainties in the proposal, but it is to be expected that there will be uncertainties at this stage of the development of a process. It is for this reason that pilot plant work is undertaken – it permits demonstration of any steps that are untested. It also permits quantification of the consumption of reagents and ensures that the composition of streams containing contaminants can be measured. The pilot plant has been reasonably professionally designed, and should yield the expected results as well as permitting some modifications should these prove necessary.

It is not possible at this stage of the development to express a firm opinion about the economics of the process, primarily because the capital cost of any full scale plant is presently a little too uncertain. On what has been presented, we are confident that the pilot plant work should clear up any uncertainties to a level sufficient to enable the full-scale plant to be designed and costed with confidence. It is, however, true to say that if the pilot plant operates close to specification, then it seems unlikely that the capital cost of a full scale plant will inflate greatly over present estimates, which would indicate a profitable process.

Appendix: Qualifications of IPC

The company has as its mission the provision of quality services to the process industries in the widest sense. Thus we have consulted to the petroleum, petrochemical, fine chemical, pharmaceutical, chemical, food processing and extractive metallurgical industries during our existence.

The company was formed in 1956, but took on its present form in 1987, when it was a subsidiary of Ed L.Bateman. It provided a range of backup services to the Bateman-Davy Limited team

which was building Mossgas, the PetroSA oil from gas plant at Mossel Bay. It then expanded its work to undertake a range of technoeconomic studies, in particular the concept engineering that led to the conversion of Middelburg Steel into Columbus; the feasibility of what became the Alusaf Hillside smelter; and a petrochemicals from coal plant based on direct coal liquefaction which has not yet become a reality.

In 1994 it became apparent that there would soon be excessive international competition in the field in which we had specialized, and Bateman opted to sell the company to the present Managing Director, Dr Philip Lloyd. Two other Directors, Ray Swanepoel and Bob Weston, agreed to stay close to the company, and continue to this day.

Philip specializes in forensic work, and has undertaken studies as wide ranging as an investigation into a metal powder blast apparently caused by the use of a non-sparking impactor; a gas explosion caused by conversion from town gas to liquid petroleum gas, and subsequent weakening of many joints in the distribution system; the preparation of a bankable feasibility for the purchase of a significant but minority shareholding in a major petroleum refinery; and has served as expert technical witness before the International Court of Arbitration regarding a turnkey project which failed to operate to specification, resulting in a $300 million claim. This project is of particular relevance to the KNEW study, as it involved ion exchange cleanup of the feed solution followed by controlled precipitation of an alkaline-earth hydroxide from a saline solution.

Ray specializes in oil refinery and petrochemical work, and has had particular success with RefModl, a refinery modeling and costing programme which has proved immensely valuable in comparing various technologies for fuel upgrading and comparative costing of those alternatives. Bob has had a significant role in resolving failed projects, winning as much value as possible for shareholders either by rectifying the problems or by finding ways of recovering some value from the assets.

The company is based in Cape Town and has a presence in Johannesburg. It does not compete on rates, but prefers to assure its clients of quality services delivered in the shortest possible time. It has a wide range of associates to which it can turn to respond to questions of the edges of its existing expertise.

CURRICULUM VITAE

NAME: Philip Lloyd

YEAR OF BIRTH: 1936

TITLE: Managing Director, IPC

NATIONALITY: South African

MARITAL STATUS: Married

QUALIFICATIONS: Pr.Eng., B.Sc.(Chem. Eng.), Ph.D.,

FSAAE, FSAIChE, FSAIMM, MSACI

LANGUAGES: Afrikaans

Working knowledge of German

GENERAL

I am a senior engineer with a wide range of industrial experience in both contracting and consulting. I also have returned to academic life and have spent much of the past 15 years in many aspects of energy. Industrially, in recent years I have been deeply involved as the expert witness for the plaintiff regarding a major minerals processing plant which failed, before the International Court of Arbitration, and in the development of a billion dollar energy project in Southern Africa. Academically I have been concerned with climate change, carbon capture and storage, and means for energising lower income homes.

EXPERIENCE

1988 - Present **INDUSTRIAL & PETROCHEMICAL CONSULTANTS**

Managing Director

Responsible for pre-feasibility, feasibility, environmental and risk management studies on a wide range of process industry projects. Recent studies have included risk management of the commissioning of Majuba power station; an assessment of a US$2billion petroleum refinery project in SA for a major bank; recommendations for the startup of a €44 million acid plant that was functionally inoperable; a report on the future liquid fuels supply in SA for the Department of Minerals & Energy, including an assessment of biofuel supply, demand and qualities; acting as an expert witness in a major water pollution matter in Vanderbijlpark; a study into markets for 1tcf of natural gas; acting as the expert technical witness for the owner before the International Court of Arbitration in a $300 million claim regarding a failed turnkey project; a feasibility study underpinning bankable documentation for a P7 billion project in Botswana, in association with a team from Royal Dutch Shell, Hague Head Office; and representation of South Africa at the International Atomic Energy Agency, working group on indicators for nuclear power. I have also acted as chairman of technical committees developing the standards for paraffin stoves, SANS 1906 and 1243, and of the Voluntary Public Affairs committee of the Chemical and Allied Industries Association, concerned with the implementation of the Responsible Care programme, particularly concerned with the transport and storage of hazardous chemicals. I also serve on the Engineering Council's committee developing means to identify work of a nature that should be reserved for engineers.

2009 – Present **CAPE PENINSULA UNIVERSITY OF TECHNOLOGY**

Professor, Energy Institute

Studies on the use of paraffin in households; on carbon capture and storage; on energy usage in low-income homes; on the dynamics of rainfall and the chemistry of

precipitation; the development of a hybrid photovoltaic system designed to provide high reliability under conditions of regular deep drawdown of storage batteries; the release of methane from coal mines; the impact of solar water heaters in entry-level homes; energy efficiency in commercial buildings.

1999 – 2009 **UNIVERSITY OF CAPE TOWN**

Senior Research Fellow, Energy Research Centre

Responsible for, or part of team, involved in studies including the Integrated Energy Plan for South Africa; evaluation of the national baseline of GHG emissions by the coal mining industry, and mitigation of those emissions; the intervention potential for low-smoke fuel in the household coal distribution chain; an evaluation of coal discards and the potential for their wider use; the use and abuse of liquid and gaseous fuels in low-income households; macroeconomic reform and sustainable development in the liquid fuels industry in SA; and the evaluation of the CDM potential of a range of renewable options including solar-thermal, photovoltaic and micro-hydro. Work included being a Co-ordinating Lead Author in the Technical Report on carbon capture and storage for the IPCC; and identification of methods for the reliable detection of trends in environmental data.

1994- 9 **UNIVERSITY OF THE WITWATERSRAND**
Professor, School of Process and Materials Engineering

Research and teaching environmental chemical engineering including HAZOPs, Responsible Care and responsibilities under Occupational Health & Safety Act and National Environmental Management Act.

1987-8 **GROUP TECHNOLOGY MANAGER, Ed.L BATEMAN**

Responsible for the acquisition and maintenance of technologies across the entire Group, including operations in Europe, Australasia and North and South America.

1983-7 **MANAGING DIRECTOR, EMS MINERALS, SUBSIDIARY OF MURRAY & ROBERTS**

Responsible for sales and the execution of work by a staff of up to 1250 on a variety of projects in the minerals industry, including Richards Bay Coal Terminal Phase 3, a wide range of cooling systems for mines, underground handling systems including long conveyor systems, several shafts, a uranium process plant for the Atomic Energy Board, a coal washing plant for Leeuwfontein, coal supply systems for Kendal and Matimba power stations; and head-end design for Mossgas, the world's first gas-to-liquids plant.

1973-83 **DIRECTOR, METALLURGY, CHAMBER OF MINES RESEARCH ORGANISATION**

Responsible for development projects over a wide range of interest to the gold mining industry, including backfill and means for its production, valuation of mines using radio-isotope sampling methods on the working face, carbon-in-pulp recovery of gold, studies of mine call factors, gravity and flotation concentration of gold. Involved in both gold and uranium marketing. Responsibilities included management of a staff of up to 200.

1967-73 **DIRECTOR, PHYSICAL SCIENCES, CHAMBER OF MINES RESEARCH ORGANISATION**

Responsible for resolution of a range of mining industry problems, including vegetation of dumps, control of dust, timber decay and its health effects, fire control methods, radiation control, and radiation monitoring. Also responsible for a major project on the recovery of uranium from slimes dams, which led in due course to the establishment of ERGO and other similar projects. Staff of up to 60.

1965-7 **SENIOR RESEARCH OFFICER, ATOMIC ENERGY BOARD**

Extraction Metallurgy Division

Upgrading of uranium ore to nuclear specifications, production of uranium metal, production of uranium hexafluoride; alternative enrichment methods; reactor engineering.

1962-5 **FELLOW, SCHOOL FOR ADVANCED STUDIES, MIT**

Studies in Nuclear Engineering, including uranium enrichment by novel methods and nuclear fuel reprocessing.

1961-2 **RESEARCH OFFICER, ATOMIC ENERGY BOARD**

Extraction Metallurgy Division at National Institute for Metallurgy

Responsible for development of Purlex process (subject of PhD) and its introduction into the uranium industry.

AWARDS

Awards include the gold medal of the SA Institution of Mining & Metallurgy; one of Four Outstanding Young South Africans by the Jaycees; several academic fellowships; in 2007, for the best paper at the International Conference on the Domestic Use of Energy; citation by the IPCC for work leading to the award of the Nobel Peace Prize jointly with Al Gore; and citation as Energy Man-of-the-Year 2010 by the SA National Energy Association.

PROFESSIONAL

Past President, SAIChE; FSPE; AS&TS. Served on Exco of SACPE for 15 years, and on many of SACPE's and ECSA's committees; SA representative at the Commonwealth Engineering Council, London, and World Federation of Engineering Organisations, Warsaw, 1977 and COP17, Durban 2011; on the International Committee for Solvent Extraction, and as Chairman of the International Solvent Extraction Conference 2002; on the National Chemical Research Laboratory Advisory Committee; on the Mintek

Advisory Committee; on several SABS Advisory Committees; Chair of Public Affairs Forum of the Chemical and Allied Industries Association; and on the Advisory Committee of the SA Centre for Carbon Capture and Storage. Over 100 publications in the professional and technical press.

PUBLICATIONS

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