Carbon Cycle Ltd

ERA Grand Challenge Round 1

Non-Confidential Final Report

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1. EXECUTIVE SUMMARY

The aim of the ERA funded project was to progress development of the Carbon Cycle process through to the completion of a Front End Engineering Design (FEED) study and, whilst we have not achieved this in totality, we have made significant progress that will enable this to happen. We were held up by, but have been able to resolve, two issues fundamental to the economics of the process. These frustrated our progress and meant we had to halt the FEED study which was approximately 70% complete. The first issue we encountered was the requirement to purify the gypsum feedstock so that high value PCC can be produced. Whilst we realised from the outset of the project that this was key, we didn't appreciate how difficult this task would be. We have learned that if the gypsum is not clean the PCC produced has little inherent value and without high value PCC the process is not financially viable. Without a cost effective method of achieving gypsum clean up any project based on this reaction is doomed to failure. We have found evidence in the literature that others have looked at cleaning up gypsum in the past but have never found a viable way to achieve this.

However, after much effort we have created a practical and low cost way to purify all forms of gypsum. Examples of what we can achieve are shown in Figure 1. The gypsum clean-up, whilst being a significant problem, which held up progress, has now potentially become a significant asset. We have filed a patent application on our discovery which we believe should have real commercial value. In addition, we have already started discussions with interested parties.

The second issue, identified as the FEED study progressed, was that the capital costs of the production plant were significantly higher than had been anticipated and too high to be supported by the potential revenue. This required us to go back to the drawing board a number of times and reengineer a number of key elements of the design. Once again this frustrated progress but the end result is that we have a much simpler and more robust design with a smaller footprint. Combining the two solutions we now have a process which is economically viable, has strong intellectual property and provides environmental benefit in reduced carbon footprint for the two products produced.

Calculating the carbon footprint of the combined products produced from our process, as it currently stands, shows a reduction of about 35% below that of current production methods if our process was integrated with a waste heat source.¹ Further reductions may be possible through internal process integration which has not been properly explored yet.

Our analysis indicates that a Carbon Cycle plant in Alberta producing ammonium sulphate and PCC would be highly profitable whilst also cutting carbon dioxide emissions. Indicative internal rates of return for the operation of a commercial plant capturing 12,800 tons/year of carbon dioxide in Alberta are 29 - 41%. The pricing of the input commodities, gypsum and ammonia, in Alberta are favourable and there is strong demand for ammonium sulphate fertiliser. The process can produce both PCC and gypsum for use as white pigments and the local availability of white pigments is likely to have positive implications for a number of Alberta industries principally paper making but also paint and plastics manufacture, amongst others. Alberta has wide spread significant gypsum deposits and would be well positioned to benefit from this technology while reducing its overall carbon footprint. It has

¹ Please see appendix 7 for a summary of how this calculation was carried out.

an extensive pulp industry but only a single paper mill that produces newsprint rather than higher quality papers due to the lack of access to reasonably priced white pigments. The ability to turn local gypsums into white pigments has the potential to change this.

We believe that our process will have real benefits for Alberta and provides an important economic opportunity. Accordingly we have made a number of recommendations regarding how this project could be taken forward and supported by ERA. These are:

- Further development of a financial model for a plant in Alberta
- Funding/Support to optimise the gypsum purification and preparation processes
- Funding/Support for Integrated Scale Testing
- Funding/Support for development of Vapour Containment
- Funding/Support to Complete the FEED study

Figure 1: Starting and Purified Gypsums



2. INTRODUCTION

The aim of the ERA funded project was to progress development of the Carbon Cycle process through to the completion of a Front End Engineering Design (FEED) study and, whilst we have not achieved this in totality, we have made significant progress that will enable this to happen. Funds, originally budgeted for completion of the FEED study, were instead diverted to resolving the issues that caused it to be halted.

We have been able to resolve an issue which is fundamental to the economics of the process. This is the ability to purify the gypsum feedstock so that useful high value PCC can be produced by the process. Without high value PCC the process is not financially viable. Historically, ammonium sulfate fertiliser was produced commercially from gypsum by reacting it with ammonia and carbon dioxide (the Merseburg process) and this was a common method of production in the UK, Germany, France, India and Pakistan until the 1990's. The calcium carbonate by-product in this process was simply sold cheaply to be applied to low pH soils or became a waste product. However, the rising cost of ammonia, together with the growing availability of ammonium sulfate as a by-product from plastics and steel production, meant that the process became uneconomic and the last production plants were closed in the 1990's. The fact that we can now produce a calcium carbonate from the process that has commercial value means that the process is again viable; particularly in those countries where the ammonia price is low and gypsum is readily available. As our process also uses carbon dioxide from a waste stream there is both economic and environmental benefit to the process.

We engaged Mott MacDonald to undertake the FEED study as they had been involved in the project and understood the technology/economics. Mott MacDonald had previously undertaken a techno-economic study of the process and confirmed the technical and economic viability prior to the DECC grant being awarded so they were uniquely placed. Unfortunately they were not able to complete the FEED study as intended but significant progress was made to the point where we had to put it on hold. As the FEED study progressed it became apparent that the capital cost of the process, as originally designed, was excessive and when put into our financial model it confirmed the process was not viable at those levels. This initiated a process of review and redesign of some of the core elements of the process. It also identified that the solution we had in place for gypsum clean up, based on acid use and distillation was totally impractical from a financial perspective. Based on this we had to do two things:

- find a less expensive way of cleaning gypsum and
- redesign some of the core processes to reduce equipment costs.

We have now addressed these issues and have a much better design which has lower CAPEX and OPEX costs. The CAPEX costs have been reduced by about 50% from the initial figures. As part of the work around the FEED study, the process was extensively modelled in detail in the engineering software package, *Prosim*. This work took nearly six months but allowed us to resolve complex process flow issues around operation conditions to minimize plant size and energy input. As a result of this we were able to create a detailed energy model. The engineering and energy models will be an essential foundation that future process design work will be built upon. Considerable learning and progress was made regarding the design of the process. The work that was done on the FEED study will remain useful to a future complete FEED study that incorporates the finalized gypsum purification process. In parallel with the FEED study some DECC testing work was still being conducted relating to the design of the carbon capture unit. The cross flow configuration was not the most efficient configuration and the vapour containment element of the design did not work as efficiently as required. Rather than spend additional time trying to resolve the vapour containment issue, it was decided to go around it for our initial design and come back to it for later designs. Instead a different variation was developed using a water absorber to capture much of the ammonia slip and generate some of the required aqueous ammonia feed stock and then the remaining ammonia slip was captured using a sulphuric acid scrubber to generate additional ammonium sulfate as product. This is an effective and simple solution and even adds an incremental profit to the financials. The downside is the additional tonnes of ammonium sulfate that are produced increase the storage and handling of input and output materials making the plant somewhat larger. Similarly we have decided to go with a standard counter flow arrangement to deliver more efficient carbon dioxide capture and reduce the ammonia slip; it also gives more flexibility in the geometry of the unit to fit a desired footprint.

We have now resolved all the issues identified by both the ERA FEED study work and the DECC optimisation project and we have a viable process, both technically and financially. Ideally we would have completed the FEED study but we are in a good position to go ahead and complete it now and what we have done instead has arguably added more value. The gypsum clean-up, whilst being a significant problem holding up progress, has now become potentially a significant asset. We have filed a patent on our discovery which we believe should have real commercial value.

The following section provides detailed commentary on the ERA tasks, the work undertaken and what has been achieved.

3. DISCUSSION OF WORKSCOPE METHODS AND RESULTS

The ERA project consisted of the following tasks and these will be used to report the methods and outcomes:

- Separation of Contaminants
- PCC Separation and Washing Method
- Basic Design FEED Study Peripheral Units
- Sump Design
- Basic Plant Design Process Controls and HAZID
- Crystallization Study
- Humidity Management Adjustment
- Basic Plant Design Capture Unit

3.1. Separating of Contaminants

Purpose and Outcome

The purpose of this task was to determine the best method of separating contaminants so that we are processing 'clean' gypsum.

It's true to say that we hadn't appreciated the difficulty in cleaning up gypsum to the level required to achieve clean PCC. We naively thought that there would be a turn-key solution to this part of the project by drawing on the expertise of the mining industry and others and hence it would essentially just be a case of finding the configuration that met our requirement. However we now realise that this is a challenge that has eluded the many who have tried before.

Our process requires very high purity gypsum as a feed stock. Without high purity gypsum, it is impossible to create PCC that meets the commercial requirements for whiteness and brightness². Even quite trivial levels of impurities will ruin the colour of PCC. Such high purity gypsum does not naturally occur as a mineral and is not commercially available. Creating high purity gypsum from widely available gypsum in a practical way that can be scaled up has proven to be extremely difficult. It has required more than two years of effort and funding significantly beyond that originally budgeted. However, after much effort we have created a practical and low cost way to purify all forms of gypsum (as illustrated in Figure 1).

² Brightness is a measure of light reflectance relative to a specific white standard at a specific wavelength. In contrast, whiteness is a measure of light reflectance across all wavelengths of light comprising the full visible spectrum. Therefore, brightness represents a more narrow measurement of light reflectance than whiteness.

Method and Work Completed

Initially we attempted to purify gypsum using standard approaches to leach out contaminants such as heating with dilute to strong solutions of alkali and acid. All these attempts failed. We tried various bleaching agents that reduced or oxidized the surface coating the gypsum. This had some whitening effects but if further grinding was necessary to reduce particle size or if chemical milling occurred during the conversion to PCC, the colour degraded. Only modest improvements in the whiteness/brightness were achieved by this work.

We then sought the aid of experts in separation techniques with the view to removing nongypsum contaminants. This was done in conjunction with extensive mineralogical work to gain a detailed understanding of the mineralogy of mined Spanish gypsum. Classification via sieving, various floatation techniques, magnetic separation, and selective staged grinding were tried. All failed to produce sufficiently pure gypsum.

We tried a different approach using hot (100°C) 96% sulphuric acid to chemically dehydrate the gypsum and create calcium sulfate. This shattered the gypsum into particles of calcium sulfate of only a few microns, allowing the separation of contaminants and producing high white/bright gypsums and PCCs. The contaminant separation was somewhat troublesome as we had to allow gypsum crystals to grow for a time to achieve the best results but we could make the process work at lab scale. At the time, it looked like the only route to white/bright PCC. We spent several months working on the issues around scaling up the process to commercial scale. This is the solution that was initially progressed in the FEED study.

The equipment required for dehydration with hot sulphuric acid and the gypsum rehydration is expensive. The equipment to distil and remove the water released into the acid during the gypsum dehydration is exceedingly expensive. Equally we discovered by analysis of an array of different source gypsums that all the gypsums that we tested had small amounts of fluoride minerals present. The equipment required for the sulphuric acid handling and distilling at these temperatures is severely damaged by even 3 ppm fluoride contamination due to the formation of hydrofluoric acid. It is impossible to economically ensure that all fluoride from the feedstock is removed to such a low level. Once we understood the fluoride and cost issues, we ceased work on sulphuric acid clean-up of gypsum. The concentrated sulphuric acid process is not viable at scale.

We then attempted to build upon the learning gained by the mineralogical studies and returned to our earlier efforts to leach and whiten the gypsum and PCC. We ground gypsum to a variety of sizes below 40 microns in a stirred ball mill using developed grinding plots with and without sieving. We then tried an evolving progression of purification techniques to remove contaminants. This was done in conjunction with reductive and oxidizing whitening techniques such as hydrogen peroxide, chlorine, hydrosulphite, and persulfate. We found that oxidizing techniques worked better on the gypsum and reducing agents tended to improve the PCC as it shifted the oxidation state of iron oxide contaminants from 3+ to 2+ which shifted from a yellow colour to a blue green colour. Our results improved and we achieved brightness results for our PCC in the high eighties but this was not good enough. Commercial PCC requires brightness values of 94% or greater. Some of the results

from our extensive testing are shown in Appendix Two: Results of Earlier Brightness and Whiteness Purification Efforts.

We then tried adapting a gypsum crystallization technique we found in an old patent using phosphoric acid in conjunction with our now extensive understanding and data base of purification and whitening techniques. This involved thermally dehydrating the gypsum to shatter the crystals and release contaminates followed by rehydration with crystallization. We found we could produce gypsum that was 91% bright but the process is difficult and slow as it required time for gypsum crystals to grow and exclude contaminants. If we tried to speed up the re-crystallization process from a time of many hours, the resulting PCC quality fell sharply. The PCC that we were creating was becoming steadily better but the process was painfully slow.

It was then that we had our "eureka moment" using a process that creates double salts. The process is relatively simple to undertake. We have continued to evolve the purification process. We can achieve brightness values for produced gypsum and PCC of 97%. This will be more than acceptable for wide range of commercial applications including paper production.

Our new process avoids the use of dangerous and toxic materials. The equipment required is straight forward and does not require the use of expensive construction materials. We have made some early efforts to scale the process from lab bench to kilos of production. The process appears to be scalable. We have tested our process against even the most impure samples of gypsum. All samples that we have tested our process on can be purified using our purification method. Heating at higher temperatures tends to improve results and increases the rate of conversion. We have seen conversion times of 15 minutes at atmospheric pressure and it may be possible to achieve it even quicker if pressure is applied.

We believe the process that we are now evolving represents a viable low cost method to purify gypsum at large scale.

3.2. Define PCC Separation and Washing

Purpose and Outcome

The purpose of this task was to determine and test the best options for separation and decide which options offer the best balance for capital investment, energy use and reliability.

The main focus of this task was to be able to separate the PCC from any residue gypsum after the main process reaction. However, through test and experimentation we found that it was fully possible to control the reaction conditions in the PCC reactor (R-2) to achieve full conversion of the input gypsum making separation of unreacted gypsum unnecessary. This simplifies the overall process. However the PCC still requires washing to remove remnants of ammonia/ammonium sulfate. The water required to do this plays a large role in the water balance of the plant. As a result this was an integral part of the FEED study as integration of the washing processes and other process stages is required to recycle water, reduce liquid waste and limit evaporation requirements.

Method and Work Completed

To reduce the creation of wash water, we looked at options for first thickening the PCC slurry to higher level of solids. Hydro-cyclones can achieve this but efficiencies decrease as particle size reduces. Our D_{50} particle size target is approximately 2.0 microns and this is below optimal operation for a hydro-cyclone but we still anticipate that a hydro-cyclone will be able to usefully thicken fluids as our design has a fluid recirculation loop (see Appendix 3, Process Overview and Process Flow Diagram of Carbon Cycle Process, labelled parts L and S). This will be fully determined during future work on scale up as part of progress towards restarting the FEED study. The thickened slurry will then passes to a centrifuge or filter press to separate fluids from the PCC. The PCC is then washed with water in a counter current washing cascade process which reduces the amount of water required. This reduces the amount and processing of the washings. Once clean PCC is achieved, it can be further dewatered in a higher speed centrifuge to create a low water product. Alternatively low water content PCC can be created using a filter press. We anticipate that dry or near dry material called crumble will not be required if the production plant is located at a paper mill. Paper mills typically use 50 to 60% by weight PCC slurries. Such slurries avoid the need for extensive dewatering and drying and are easier to use for the paper mill. Slurries represent a lower energy option for product supply as dewatering and drying is energy intensive. High solids slurries of PCC are unstable and must be produced near the point of use.

In order to create a tighter particle distribution of the produced PCC, it may be useful to use a dynamic classification mill as shown in Figure Two. If classification is done, product that is found to be either too small or too big is separated and sold to other markets such as adhesives for fine materials.

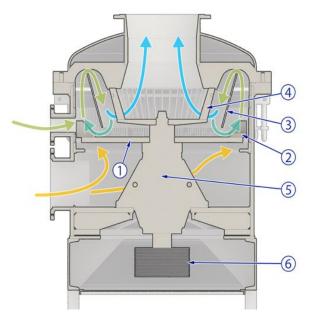


Figure Two: Dynamic Classification Mill³

³ Diagram of dynamic classification mill is taken from Atritor's web site who we have had a number of discussions with about the application of their equipment to our process. As we currently have not scaled up to the sample size required for testing we have not yet had trials done using their equipment. When we have optimized our purification and production processes, we will have trials run if appropriate.

Cross section of typical DCM showing flow of material through the mill

- Feed material inlet 1 Grinding rotor
- Main air inlet 2 Grinding zone
- Fine product and air 3 Baffle
 - 4 Classifier rotor
 - 5 Bearing housing and shafts
 - 6 Belt drive

We understand our options and best route for washing the produced PCC. We have supplier contacts and costs to undertake trials of our product when we have finalized our gypsum purification and processing work. The exact particle size of the inputted gypsum and its ratio to reaction solution directly impacts on the PCC that is created. For this reason, we can only finalize our equipment choices when we have finalized our gypsum grinding, purification and processing work. Once this is done, we will produce sufficient samples of PCC for supplier trials. These trials will give us separation efficiency, required energy input and capital costs which we will feed back into our Prosim and energy balance models. From this we will be able to choose the best options for a commercial production plant.

3.3. Basic Plant Design – Peripheral Units

Purpose and Outcome

A 'basis of design' was agreed at the outset of the study. The basis of design defined the parameters for the FEED study such as days/hours of operation, output capacity, storage capacities for inputs and outputs, the methods of material delivery and the process flow diagram.

The peripheral unit's element of the FEED study was focused on the equipment and infrastructure that supports the process and would form the framework for the core study to follow. It incorporated the following aspects of the engineering design:

- Site Layout
- Incoming feedstocks supply and storage
 - o Ammonia
 - o CO2
 - Sulphuric Acid
 - o Gypsum
- Outgoing products supply and storage
 - Ammonium Sulfate
 - o PCC
- Staff facilities including offices, toilets, catering, car parking
- Civil engineering including buildings, roads, lighting, utilities
- Waste processing and sewerage
- Ancillary process equipment
 - PCC washing
 - Ammonium Sulfate Crystallisation

Method and Work Completed

The work was largely conducted in conjunction with Mott MacDonald. A listing of the main operational plant areas and detailed documents produced as a result of this work is listed in appendix 4. The documents are available for review if required.

3.4. Sump Design

Purpose and Outcome

This task, to design and test flushing and sloping v-groove sumps, was originally required due to problems caused by having gypsum circulating in the carbon capture reactor. Gypsum has a tendency to settle and clog, and this caused a real issue in our earlier testing of the process. Hence we defined a task where we would design a sump specifically to address these issues. However the final design has resulted in the gypsum only being input to the PCC reaction vessel (R-2) and hence we have been able to avoid the problem of gypsum slurry settling in sumps. The gypsum that is added to the PCC reaction vessel (R-2) is directly reacted and forms PCC which has less settling issues compared to gypsum. This allows us to use standard shaped sumps and vessels.

Method and Work Completed

This ended up being standard equipment and was incorporated into the FEED study work.

3.5. Basic Plant Design – Process Controls and HAZID

Purpose and Outcome

This work built on the peripherals FEED study previously started to include the process controls and Hazard Identification (HAZID) study. Process control systems deal with maintaining the output of a specific process within a desired range to ensure both the quality and safety of production. The purpose of HAZID is to identify the potential hazards and to reduce the probability and consequences of an incident and its impact on the personnel, properties and environment. Whilst significant progress was made in this work, it couldn't be completed due to the changes in design and the outstanding issue of gypsum clean-up.

Method and Work Completed

Since we had better understanding, we took on the task of preparing a process controls document. This gave a generic overview of how the process would be monitored and controlled within each section of the plant. The gypsum clean-up section had to be left out as the process steps hadn't been fully defined at that time. For the HAZID study, a full day workshop was held at Mott MacDonald offices with everyone working on the project being present. We worked to identify all the possible hazards and what could be done to reduce the possibility of them occurring. Another full day workshop was conducted by only Mott MacDonald and they prepared a HAZID report. However, this report did not include the main capture unit section of the plant as it was only focused on the peripherals. The HAZID study can be reviewed in Appendix eight.

3.6. Method of Crystallizing

Purpose and Outcome

This task was to determine the best value way to create large ammonium sulfate crystals which have a higher value and a larger potential market. Initial research was conducted by Carbon Cycle and quotes sought from manufacturers. As a result of this work we moved from large crystals to fine crystallisation followed by granulation, and from cooling (thermal swing) to evaporative crystallisation. Once the equipment and costs had been established these were combined into the FEED study for incorporation in the overall design.

Method and Work Completed

Industrially there are three processes for crystallization:

- a) Cooling (thermal swing) crystallization
- b) Evaporative crystallization
- c) Reaction crystallization

Initial work was developed on using a cooling thermal swing to form the ammonium sulfate crystals from a hot supersaturated ammonium sulfate solution; as we believed this would be

the lowest energy route as there would be no energy required to evaporate water. Through discussions with equipment suppliers and technical experts on crystallisation we found however that the recycle rate required due to ammonium sulfate solubility's low sensitivity to temperature made this unfeasible, as equipment became excessively large and costly for the crystallisation rates necessary.

Reactive crystallisation relies on creating supersaturation by reaction of the material you wish to crystallise. For our process, this would mean consuming far more ammonia and sulphuric acid. Both are expensive commodities and producing correspondingly larger amounts of ammonium sulfate causing storage issues; and so this left us with evaporative crystallisation.

Evaporative crystallisation was the method recommended by the suppliers and experts. We also found that techniques including mechanical vapour recompression have been developed to recover as much energy as possible from the crystallisation to reduce the large energy input.

Once the type of crystallisation was selected we had to decide on what crystals to produce. Both fine crystal and large crystal crystallization processes are used for crystallizing ammonium sulfate. The costs, size and energy requirements for large crystal (2 to 5 mm) ammonium sulfate crystallization are substantially greater than for fine crystal ammonium sulfate. The market size and price for large crystal ammonium sulfate is much greater than exists for fine crystal ammonium sulfate. To enable us to take advantage of the lower energy and capital cost of fine crystal crystallization, we looked into the cost and energy requirements of granulation where fine crystals are mixed with either a small amount of water or a binding agent such as gypsum and compressed into granules. The combination of fine crystal crystallization. Equally granulation presents the opportunity to mix in other fertilizer nutrients such as phosphate into the granules to create tailored nutrients that will appeal to a wider market.

We asked for guide quotations for plant and design from several suppliers. Of the quotes that we received, the best option was from France Evaporation for the crystallization plant and from Sanhut Conreur for the granulation plant.

3.7. Humidity Management

Purpose and Outcome

As mentioned previously, our work under the DECC grant to develop our vapour containment process to contain ammonia and water vapour was not completed to a level that could be usefully applied to production plant design. Further work to establish key aspects of this technology will be required before this can happen. Therefore our design does not include systems for minimizing water vapour loss from the process. Primarily water and ammonia loss will occur within the carbon capture unit (R-1). Water and ammonia vapour will seek to reach equilibrium with the flowing gas. Ammonia vapour is removed from the flowing gas by reaction with sulphuric acid. Regretfully little can be done for the humidity losses from the process at this time but it is a relatively minor issue and the overall process remains viable with or without vapour containment.

Method and Work Completed

No work undertaken.

3.8. Basic Plant Design – Capture Unit

Purpose and Outcome

This purpose of this work was to build on the elements of the FEED study previously completed to include the capture unit processing and integrate all the components together.

The design of the capture unit changed fundamentally as a result of testing completed under the DECC optimisation project. Whilst we started with a crossflow capture unit with vapour containment units scrubbing and recycling the ammonia, we ended with a counter current design that absorbs the ammonia slip using water that is then used to dilute the incoming ammonia. Any slip past the water absorber is dealt with by a small acid scrubber which makes additional ammonium sulfate. These changes made the process simpler and also mean that standard equipment can be used reducing project risk and cost.

The other major change came around using gypsum in the capture unit; experimentation demonstrated that it provided no benefit to the carbon dioxide capture efficiency. This in combination with the settling and clogging issues it poses meant it was removed from the capture unit and instead added directly to the PCC reactor. This simplified the capture unit design and meant standard sump designs could be used.

Work Completed and Achievement

This work was based on testing that had previously been conducted in our various test units. Our initial design used a cross flow arrangement where a fluid slurry of water, gypsum, PCC and ammonia was pumped over fill packs and a gas such as flue gas containing increased levels of carbon dioxide (from atmospheric concentrations to greater than ten percent carbon dioxide) was passed through the fill pack perpendicularly. The modelling of the gas (carbon dioxide, ammonia and humidity levels) and aqueous phases (gypsum (dissolved and out of solution), PCC (out of solution) and, ammonium bicarbonate (dissolved)) in such an arrangement is exceedingly complicated as the concentrations are different in all parts of the pack and gas flow, varying in x-, y- and z-axis's. To create a simpler system we experimented both with co-flow and counter current designs, with the liquid and gas moving co-axially and opposingly respectively. Through experimentation it was found that counter current provided better carbon dioxide capture efficiency with similar ammonia slip and also allowed for more flexibility in the geometry of the unit. The counter current design means that the concentration of ammonia and carbon dioxide will be consistent across the exit face of the fill pack and is far more easily modelled as variation is only in one axis, rather than three of cross flow.

Our experimentation work showed that gypsum being present in the capture reactor provided no benefit to the carbon dioxide capture efficiency. When this was considered

with the many issues having gypsum in the capture unit causes due to its sticky nature e.g. settling and clogging of packing and piping connections, it meant its removal from the capture unit was the best course of action. Instead addition directly to the PCC reactor (R-2) will be utilised where settling and clogging are not such big issues.

3.9. Overall Energy Balance

Purpose and Outcome

As the foundation of the FEED study work it was necessary to develop multiple process models for the plant which provide the data necessary for designing equipment and determining costs. Several iterations of process model were necessary to identify the optimal process conditions and due to changes in the processes leading on from experimentation results, several versions of the process design had models developed. The process modelling included developing the following: process flow diagrams, preliminary piping and instrumentation diagrams, mass balances, water balances, and energy balances. These enabled us to understand the implications of process conditions and arrangements on equipment choice (type, size and cost), water usage, input/output volumes, and energy costs of different design choices. Through the use of these documents it was possible to determine our preferred process case in terms of CAPEX and OPEX; this was found to be the 2% ammonium bicarbonate in PCC Reaction Vessel (R-2) Case, further details of the case can be found in Appendix 5.

Work Completed and Achievement

The design process began with constructing the process flow diagrams for the plant. These formed the basis around which the mass, energy and water balances were created. Due to changes to process sections from developments in our experimentation the process flow diagrams went through several iterations and therefore so did the balance calculations. Once the design was more settled, preliminary design of piping and instrumentation diagrams was conducted in partnership with Mott McDonald. These were not fully completed due to the halting of the FEED study but the preliminary versions provide a base for their future development. Through iterative versions of all the process design documents, we narrowed down the conditions and cycles to a reduced number of potential scenario cases. These final cases were created as complex process model simulation within ProSim Plus, a chemical engineering modelling software package. The scenarios modelled in the package are outlined in Appendix Five. This allowed us to determine that the optimal scenarios in terms of energy input using lower ammonium bicarbonate (AB) concentrations in the PCC reactor which in turn lowered the large energy input to the AB remover and raised the concentration of ammonium sulfate fed to crystallisation reducing the energy to evaporate, the other large energy input. Thus scenarios running at 0% AB and 2% AB in the carbon capture unit (R-1) where carbon dioxide is captured represented the best cases. As the energy input difference was minimal between the two cases, but the size and so cost of the PCC reactor would be much greater due to an much increased residence time from running AB concentration down to zero for the 0% case, it was determined that the 2% AB scenario represented the best case in terms of both CAPEX and OPEX.

Further refinement of the model for the exact equipment selection based on supplier information was not possible as the FEED study was halted prior to this point, however

the simulations created are in position to be updated and refined with this information when it becomes available.

4. THE ECOMOMIC CASE FOR A PLANT IN ALBERTA

The markets for ammonia and ammonium sulfate vary wildly by region. Britain has probably the most distorted nitrogen markets in the world due to the near total dominance of a single large company in the market. Ammonia costs are up to 50% higher than in other parts of the world. At the same time ammonium sulfate prices can be less than the value of the ammonia content of the ammonium sulfate. Alberta has a very different situation. Ammonia costs are some of the most competitive in the world. Multiple actors are present in the ammonia and ammonium sulfate markets. There is a strong developed market for ammonium sulfate due to many of Alberta's soils and crops requiring sulphur addition as fertiliser. In Alberta, our process would be viable and give strong investment returns.

A commercial scale production plant that captures 12,800 tons of carbon dioxide per year from a gas combustion stream of 3% carbon dioxide will produce 60,448 tons of ammonium sulfate and 29,120 tons of PCC. The operation of this plant is expected to have a lower limit 10 year IRR of 29.1% and an ROE of 49.1%. These returns assume a predicted upper limit for plant build costs of twenty million pounds (sterling). Our target build cost is sixteen million pounds (sterling) and looks achievable.

Alberta has strong demand for ammonium sulfate as many of its soils have had the sulfate leached out. In Alberta, sulphur is the third most required soil nutrient after nitrogen and phosphate. Many of the crops grown in Alberta need sulphur addition in order to achieve good yields. Canola is a good example of this. Alberta does not have a sufficient internal production of ammonium sulfate and is forced to import ammonium sulfate⁴. This negates the advantage that Alberta has from its access to low cost ammonia supply. The majority of the world supply of ammonium sulfate is as a by-product from plastics and steel production. Alberta imports substantial volumes of ammonium sulfate. Across Canada, approximately 10.5% of fertilizer sales are ammonium sulfate but across the Western Prairies, consumption is higher⁵.

The carbon footprint of the combined products produced from our process is expected to be 30% below that of current production methods. If our process was integrated with a waste heat source, the production carbon footprint could fall by 40% or more. Further reductions may be possible through internal process integration which has not been properly explored yet.

The markets for PCC and GCC cover a wide range of applications and a wide range of prices. Our model uses PCC prices that we have seen in Europe which has access to white pigments feed stocks that are relatively close to the sea. In Alberta, pigment and pigment feed stocks have to travel more than 1000 miles by land. This is expected to raise costs. *Our economic model does not include the potential price premium that we think will exist in Alberta for PCC/GCC. We have used European PCC prices that are likely to be lower than Alberta.*

⁴ We could only find 350K tons at Redwater and 70K tons at Fort Saskatchewan. A discussion of the requirement to import ammonium sulphate into Alberta can be found at

http://www.agcanada.com/daily/ammonium-sulphate-seen-tougher-to-find-afford

⁵ Canadian Farm Fuel and Fertilizer: Prices and Expenses (July 2015), Figure 5

Our financial model is attached to this report as a spread sheet model entitled, "CCL Financial Model Alberta". The predicted profit statement for the plant is shown in Appendix six. The model looks at the economic viability of a commercial scale production plant that captures 12,800 tons of carbon dioxide from a 3% gas stream and produces ammonium sulfate and PCC. The model currently calculates costs in British pounds. The model uses costs that we have found for Alberta's ammonium sulfate. ⁶ It uses labour and typical European overhead costs. ⁷ While Alberta's costs will be different, the model gives a reasonable indication of the commercial potential of our process. Using a low price for PCC as slurry at a paper mill (£225/ ton)⁸ indicates investment returns of IRR (10 year) of 29.1% and ROE of 49.1%. If our process produced dry PCC which has higher costs,⁹ indicative investment returns are 10 year IRR of 41.2% and ROE of 75.4%. Our financial model values carbon credits at zero.

⁶ We used the lower ammonium sulphate cost \$470 to reflect seasonal movement from the discussion <u>http://www.agcanada.com/daily/ammonium-sulphate-seen-tougher-to-find-afford</u> and reduced the price by 25% to allow for distributor costs. Converted into pounds this is £211/ton. We used the Gulf cost for ammonia at the same time as the prices quoted in the ammonium sulphate discussion. We suspect that Alberta's wholesale ammonia costs are lower but we do not have market data for the time in question to substantiate this. Hence we used the Gulf price of \$385/ton FOB NOLA, 22 Oct 2015, ICIS Fertilizer, The Market, p17. 1 CAD = £0.60

⁷ We would welcome incubation support to create a more detailed model reflecting Alberta's specific cost structures.

⁸ This cost reflects discussions we have had with Scottish paper mills. It is likely to be on the low side for PCC. Distributers have indicated higher prices.

⁹ In Britain we are told we would expect to see market prices of £400 to £450 per ton for dry PCC. Allowing for distributor costs, we have assumed a dry PCC factory gate price of £320 per ton PCC.

5: CARBON FOOTPRINT

Our process has a lower carbon footprint for the production of ammonium sulphate and PCC than current production methods. We calculated two carbon footprint examples for Alberta, operating our process at a paper mill and at an ammonia production plant. The detailed calculations for the carbon footprints and energy balances are shown in Appendix seven. Appendix seven also include process flow diagrams that show the carbon dioxide emitted by the process vs the carbon dioxide converted by the process.

Figure Three: Carbon Footprints for Alberta Production

| Process | Carbon Footprint* | Percentage Reduction | | | | |
|----------------------------|-------------------|----------------------|--|--|--|--|
| Published Industry Figures | 3.23 | ~ | | | | |
| Alberta Ammonia Plant | 2.29 | 29.1% | | | | |
| Alberta paper Plant | 2.11 | 34.6% | | | | |

Carbon foot print is for plant that captures 40 tons per day of carbon dioxide and makes products from it.

*The carbon foot print is for every ton of carbon dioxide reacted which produces 4.79 tons of ammonium sulfate and 2.27 tons of PCC. The production of ammonium sulphate is 3 tons from the carbon capture reaction and 1.79 tons from the reaction of sulfuric acid to scrub the ammonia slip.

These numbers do not reflect potential process integration where excess heat from one part of our production process is moved to parts that need heat. Work on this is planned in the future. It is likely that future process integration will further reduce the carbon footprint.

The calculated footprints assume that gypsum is being imported from British Columbia. Opening a local quarry will eliminate the carbon release associated with the considerable distance that the gypsum is travelling. There is a relative abundance of gypsum in many parts of Alberta.

We have used published industry figures for the production of ammonium sulfate and PCC. We do not have carbon footprint figures for Alberta production of ammonium sulfate. There is no PCC production anywhere close to or in Alberta.

We have not done a life cycle calculation as we lack a completed FEED study to base this work on. Equally, we would have no figures to compare this against as we have only been able to find calculated carbon footprints and not life cycle carbon numbers for current methods of product production.

6. CONCLUSIONS

Carbon Cycle has successfully created a new low cost and low energy method of purifying gypsum to create high purity gypsum from a variety of plentiful low cost sources. The ability to purify gypsum is critical to making the Carbon Cycle process economically viable. Without it, the value of the PCC that our process produces would be worth 5 to 10% of PCC that meets market required properties. In the effort to find a method to purify gypsum all other published processes were reviewed and failed to be viable/successful to purify gypsum at scale. Based upon current work being undertaken, our purification method is anticipated to have low CAPEX and OPEX costs. The process uses safe low cost chemistries that do not interfere with our process's main carbon capture and production cycle reactions.

Our process represents a lower carbon footprint route to creating ammonium sulfate and PCC compared to current production methods. Without further process integration and development, we anticipate our process will reduce the carbon footprint for PCC and ammonium sulfate by 30%. If we integrated our process with a low temperature waste heat source, our process would reduce the carbon footprint for PCC and ammonium sulfate by 40% or more¹⁰.

A better understanding of commercial scale design has been gained. From this has flowed a better understanding of CAPEX and OPEX costs. Based upon the market research we have done of required feed stocks and market prices of outputs, the process continues to have strong economics in Alberta. *Indicative rates of return for the operation of a commercial plant capturing 12,800 tons of carbon dioxide in Alberta are 29 - 41% IRR (10 year).*

Our process has split the carbon capture chemistry from the product creation chemistry in order to gain better control over the production conditions of the PCC. Equally the separation of the chemistries allows us to eliminate gypsum settling issues within the capture sump. This will allow us to use standard shaped sumps and reduce associated costs.

The unanticipated very large effort required to find a viable and scalable solution to the gypsum purification problem consumed a large amount of the project's budget. It also caused the project to take longer than originally planned and needed to pull in funding from other sources. As a result, the delays to finding a solution to the purification study and the cost pressures that the unexpected effort required, the commercial production plant FEED study was paused half way through. The paused work remains useful and viable. It will be built upon when the FEED study is restarted.

¹⁰ Please see appendix 7 for a summary of how this calculation was carried out

7. RECOMMENDATIONS

As outlined in section 4, The Economic Case for a Plant in Alberta, the Carbon Cycle process is a good fit with Alberta. Building a plant in Alberta would provide a means of capturing carbon dioxide whilst at the same time providing an opportunity for a new industry that would grow the local economy and reduce reliance on imports. For this reason we would encourage ERA to continue to support us in the development of the process. To this end we are making a number of recommendations as follows:

The development of a financial model for a plant in Alberta

A specific financial model relating to Alberta for our process should be developed. Our current model will be relatively easy to adapt for this but we need help to understand the costs of doing business in Alberta. Equally access to a report on the ammonia and ammonium sulfate markets for Alberta would be extremely helpful. Gaining a better understanding of Alberta's gypsum supply costs would also be necessary. Our model uses European costs. Support to fill in our knowledge gaps and create a financial model that is specific to Alberta is requested under incubation support.

Funding/Support to optimise the gypsum purification and preparation processes

Currently Carbon Cycle is working on optimizing our gypsum purification and preparation processes to reduce inputted energy and CAPEX costs. This will be done by minimizing the grinding of the gypsum and the cycle time for the gypsum purification while producing fine particle PCC with a tight distribution curve. This work will require controlled grinding of the gypsum feed stock, purification and then producing PCC while continuously measuring particle size distribution. Funding and support to undertake this work is requested.

Funding/Support for Integrated Scale Testing

Once the gypsum purification and preparation process is optimized, we will undertake scale testing of the integrated process. This is required as the process has changed significantly from that initially proposed. Once this is complete, we will then be in a position to restart the FEED study for a commercial production plant using our process. Funding and support to undertake this work is requested.

Funding/Support for development of Vapour Containment

We decided to take a pragmatic approach with regards to vapour containment for our first plant rather than undertake further research and hence our current plant design does not seek to contain ammonia vapour. As a result, the released ammonia vapour has to be captured by reaction with sulphuric acid creating an additional tonnage of ammonium sulfate. Ammonium sulfate produced this way is more expensive to produce than ammonium sulfate produced from gypsum. Finishing off the development of our vapour containment system would allow us to greatly reduce or eliminate the ammonia slip from our carbon capture process and improve the viability of our process. Funding and support to undertake this work is requested.

Funding/Support to Complete the FEED study

We believe we will be in an excellent position to build upon the work already completed and finish the FEED study. We now have the technical issues resolved and a better understanding of the costs making us confident that the process can be implemented and be commercially viable.

8. ACKNOWLEDGEMENTS

We would like to thank the Canadian Province of Alberta for the grant support that made this work possible. We would also like to thank the British Department of Energy whose earlier grant laid the foundations for the work in this report.

We would like to thank Richard Nelson of Alberta Innovates, who put up with far too many questions from us and for being patient and understanding when the project overran due to the problems we encountered purifying the gypsum.

Appendixes

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Appendix One: Carbon Cycle Technology and Alberta

Carbon Cycle is working to commercialize a process that reacts carbon dioxide with ammonia and gypsum to produce ammonium sulfate, a fertilizer that supplies nitrogen and sulphur and precipitated calcium carbonate (PCC), a white pigment and filler. The markets for PCC and ammonium sulfate are large and global with volumes measured in the tens of millions of tons and billions of dollars per annum.

The overall reaction that lies at the heart of our process is:

Figure 4: Reaction Chemistry

The reaction is energetically favoured and drives forward if the conditions are suitable. The reaction takes place at carbon dioxide levels below that present in air and up to concentrations greater than that found in coal fired power plant emissions (13.5% carbon dioxide). The reaction mineralizes and stores carbon dioxide for geological time periods. The majority of carbon dioxide on Earth is stored as carbonates and most of it is as calcium carbonate. Creating calcium carbonates is a very safe and long term way to store carbon dioxide. The production reaction produces 2.27 tons of PCC and three tons of ammonium sulfate for every ton of carbon dioxide from gas streams at 3% carbon dioxide which is the typical emission stream from a gas fired electrical turbine.

The reaction products from our production reaction are easily separated. Ammonium sulfate is highly water soluble and can be crystallized out in pure form. Precipitated calcium carbonate (PCC) is insoluble under reaction conditions. If the reaction sequence is taken to completion in a polishing step, the PCC does not have to be separated from the starting reactant gypsum as all the gypsum is converted to PCC. The production reaction is well suited to scaling to commercial scale.

Ammonium sulfate is a fertilizer that provides nitrogen and sulphur. Many of Alberta's soils and grown crops require the addition of a sulphur based fertilizer. Ammonium sulfate is sold as either a near saturated solution for liquid spreading, as fine crystal or as large crystals/granules. Alberta's climate and wide spread farming area do not favour the use of liquid products. Most fertilizers are spread as large crystals/granules. Ammonium sulfate granules are less expensive to produce and allow blending with other fertilizers such as phosphate to give tailored fertilizer blends. Crystallizing ammonium sulfate requires the evaporation of water from saturated solutions of ammonium sulfate and this inevitably carries a substantial energy input penalty. The lowest energy method to crystallize ammonium sulfate is using vapour recompression. Ammonium sulfate is a fertilizer that is applied on farm during the spring. This means that a production plant will require substantial product storage during the times of the year when there is no on farm product requirement. Product prices move seasonally with prices peaking in the spring and falling to the annual minimum immediately after the fertilizer application season finishes. Product sales at reduced prices (approximately 10 to 15% below annual peak) continue during the off season to farms with storage. Ammonium sulfate prices also move with the price of ammonia which is impacted by the price of natural gas and production capacity.

Precipitated calcium carbonate (PCC) is sold for approximately one hundred different applications. Examples are: paper making, adhesives, plastics, sealants. It is primarily used a white pigment, as a filler and, for its rheological and crystal structure properties. Approximately seventy percent of production is used for paper making where it is used as a low cost filler, brightener, opacifier, and bulking agent that helps in the reduction of wood pulp and other additives in the paper manufacturing process. PCC is prized for its brightness/whiteness, defined crystal structure and, low abrasiveness due to low silica content. It often shares its markets with ground calcium carbonate (GCC) which has a lower cost but lacks a controlled crystal structure and is abrasive due to silica content. Both GCC and PCC require very high purity chalk/limestone and marble feed stocks that are not widely present and are expensive. In North America, high purity feed stocks are only found in a few places near the coasts and in Texas. PCC and GCC require high purity feed stocks.

To make PCC requires heating the feed stock to approximately 1000^oC and chemically decomposing the calcium carbonate to calcium oxide which is then cooled and mixed with water and carbon dioxide under controlled conditions to form PCC. It is a high energy input process. Equally the production of GCC is quite energy intensive. The required energy to grind fine powders rises very sharply below 12 microns.

Ammonium sulfate is rarely produced in itself. It tends to be created as a by-product of plastics molecule synthesis and from the production of steel making coke. This leads to mismatches of places of demand and places of production. A good example of this is Alberta having a strong demand for ammonium sulfate and having to import significant amounts of ammonium sulfate in spite of having large ammonia production.

The Carbon Cycle Process has a number of strategic advantages over current production methods for ammonium sulfate and PCC while offering a technology capable of capturing carbon dioxide from coal and gas fired power plants. Our process generally operates at ambient temperatures and the highest process temperature required is 900°C less than required to make PCC. Not surprisingly our process's carbon footprint is lower (30%) and with further work we think process integration can significantly improve this. Access to low temperature waste heat greater than 50°C will reduce the carbon foot print by 40% or more.

For Alberta, our process has strategic advantages that go beyond carbon reduction. Our process offers the opportunity to domestically meet the province's agricultural requirement for ammonium sulfate while using locally quarried gypsum. This will provide maximum job

creation and economic impact. Equally creating a supply of low cost, high purity PCC in Alberta far removed from existing supplies of PCC is expected to have longer term transformative effects on the paper and pulp industry of the region. Other regional potential users of PCC such as structural plastics producers are expected to benefit. Our process when developed will allow Alberta to further build upon its strategic advantages of having a large ammonia industry and a substantial forestry products industry.

| | | | | | | 1 | 1 | 1 | | 1 | 1 |
|---------------------------|--------------------------|--------------------|-----------------------|--------------------|------------------------|----------------------------|--------------------|---------------------|---------------------|------------------|----------------|
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| | | | | | | | | | | | |
| ogether | Dehydrated @ 250C | | Gypsum Ground with 5% | Sulphuric Acid | WSB-2 Meter | PCC Conditions: 1L Sol, 59 | % NH3, 5% AB | Samples Dried @ 100 | ,C | | |
| | | | | | | | | | | | |
| | | | | | | | | | | | |
| Sulphuric Acid 🛛 🔽 | Hydrogen Peroxide 🔽 | Oxalic Acid | Sodium Hypochlorite 🔽 | Descaler | Hydrazine Sulphate | Sodium Hydrosulfite 💌 | Sodium Bisulfite 🔍 | Waller's Solution | Gypsum Brightness | PCC Brightness | PCC Whitness |
| L. Yes (25%, 95°C, 2hrs) | | No | No | No | No | No | No | No | Furnace (8) | | |
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| - D | 1. Yes (15%, 3hrs) | 1. Yes (15%, 3hrs) | No | No | | | No | 2. Yes (16hrs) | 72.3 | | |
| 0 | No | No | No | No | No | | No | No | 72.3 | | |
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| . Yes (5%, 16hrs) | No | No | No | No | No | No | No | No | 77.7 | | |
| . Yes (5%, 16hrs) | 2. Yes (10%, 3hrs) | No | No | No | No | No | No | No | 83.9 | | |
| Yes (10%, 3hrs) | No | No | | No | | | No | No | 66.0 | | |
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| Yes (5%) 2. 90% | No | No | 1. Yes (1%, 16hrs) | No | No | | No | No | 75.2 | | |
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| . Yes (5%, 16hrs) | No | | 1. Yes (1%, 16hrs) | No | No | | No | No | | | |
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| Yes (1.5%, 3hrs) | No | No | 1. Yes (1%, 3hrs) | No | | No | No | No | 79.8 | | |
| . Yes (1.5%, 3hrs) | No | No | 1. Yes (1%, 3hrs) | 2. Yes (10%, 1hrs) | No | No | No | No | 63.8 | | |
| Yes (1.5%) 3. 5% | No | No | 1. Yes (1%, 3hrs) | 2. Yes (10%, 1hrs) | | No | No | No | 76.7 | | |
| Yes (3%, 3hrs) | No | No | | | | No | No | No | 78.9 | | |
| . Yes (80%, 2hrs) | No | No | | No | | | No | No | 79.3 | | |
| | No | No | | | | | | | 80.0 | 80.2 | |
| Yes (3%, 3hrs) | | | 1. Yes (0.5%, 3hrs) | No | No | No | No | No | 80.0 | | |
| Yes (5%, 2hrs) | No | No | 1. Yes (0.375%, 2hrs) | No | No | No | No | No | <u> </u> | 79.6 | |
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| Yes (96%, 2hrs) | No | No | | No | No | No | No | No | 72.6 | | |
| Yes (96%, 2hrs) | No | No | No | No | No | No | No | No | 65.4 | 69.9 | |
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| | | | | | | | | | | | |
| | | | | | | | | | | | |
| | | | | | | | | Other | | | |
| | | | | | | | | Туре | Gypsum Brightness 💌 | PCC Brightness 💌 | PCC Whitness 💌 |
| | | | | | | | 1 | Superfine White | 79.6 | | 96.2 |
| | | | | | | | 1 | Analytical Grade | 97.1 | 1 | 50.2 |
| | | | | | | | | Rakem | n/a | 97.1 | L 101.4 |
| | | | | | | | | Nakem | 11/0 | 97.1 | 101.4 |
| | | | | | | | | | | | |
| | | | | | | | | | | | |
| | Gypsum Brightness 💌 | Gypsum (g) | Solution (L) | AB (g) | NH3 (%) | AS (g) | Polymer (g) | PCC Brightness | | | |
| | 77.8 | 100 | 1 | 53 (5%) | 5 | 0 | 0 | 69.5 | | | |
| | 77.8 | 100 | | 136 (12%) | 5 | 0 | | | | | |
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| | | | | | 5 | | | | 1 | | - |
| | 75.0 | 40 | 1 | 53 (5%) | 5 | 0 | 0 | 67.1 | 4 | | |
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| | | | | | | | | | | | |

Appendix Two: Results of Earlier Brightness and Whiteness Purification Efforts

Appendix Three: Process Overview and Process Flow Diagram of Carbon Cycle Process

PROCESS OVERVIEW

Our process can be broken down into four stages:

- **1) Processing and purification of gypsum into reaction ready state** This involves grinding the gypsum initially and then purifying it.
- Carbon capture from waste gas stream
 Ninety percent of the carbon dioxide from the waste gas stream is reacted with ammonia to produce ammonium bicarbonate solution.
- 3) Reaction of ammonium carbonate with gypsum to produce ammonium sulfate and precipitated calcium carbonate (PCC)
 A concentrated solution of ammonium sulfate is created while producing insoluble PCC.
- **4)** Separation of PCC and crystallization of ammonium sulfate The PCC is separated from the ammonium sulfate and washed to recover tramp ammonium sulfate. Ammonium sulfate is concentrated and then crystallized as fine crystals and then granulated.

To reduce risk and to gain a greater degree of control over the formation conditions of PCC, we moved away from direct reaction of gypsum, ammonia and carbon dioxide (figure 4) and moved to a two stage reaction process (stages two and three). This has the added advantage that we greatly reduce potential clogging within the fill packs where the carbon dioxide is captured. It also means that we can use standard shaped sumps as we do not have to address gypsum settling during the carbon capture stage. Fine particle gypsum slurries tend to settle and form thixotropy masses unless very carefully designed equipment geometry with considerable agitation is used. Our solution sidesteps this issue.

Our current process design does not include ammonia vapour containment. This means that there is a greater than originally anticipated slip of ammonia from the carbon capture section. The released ammonia vapour is captured by reaction with sulphuric acid to produce further ammonium sulfate. We had expected to be able to apply knowledge gained from our work under the UK Department of Energy and Climate regarding development of our vapour containment system. Regretfully, we were forced to cannibalize resources from that grant that we would have applied to this to aid in the solution of the gypsum purification problem. Our proposed vapour containment system will require further development work before it can be applied to our process. Our process would benefit from having ammonia vapour containment but it is still viable without it.

Figure five shows a process diagram of our process. The diagram does not include the purification and preparation of the gypsum or the scrubbing of gas stream leaving the carbon capture section to remove ammonia vapour. In R-1, carbon dioxide from an emission source gas stream is reacted with ammonia and water flowing down a filter pack to create high surface area. The gas stream rises through the fill pack. Ninety percent of the

carbon dioxide in the gas stream is reacted and forms ammonium bicarbonate. This leaves the liquid recirculation loop via D and goes to R-2 where the ammonium bicarbonate solution is reacted with gypsum and further ammonia. This creates ammonium sulfate which remains in solution and PCC which is insoluble. The resulting slurry leaves R-2 via E and passes to a centrifuge S-1 which separates the PCC from the ammonium sulfate solution. The PCC is passed to W-1 where it is washed with water and clean PCC exits at P. Some of the separated ammonium sulfate solution is passed back to R-2. Some is transferred to M where the process stream splits. Some of the fluid is passed back to R-1 to form more ammonium bicarbonate. Some is passed to B-1 where the solution is heated to decompose and release the unreacted ammonia and ammonium bicarbonate. The heating process evaporates some water so that a greater than 40% solution of ammonium sulfate is created. Mechanical recompression may be used to aid this depending upon the cost of the local electricity. This solution is then passed to the crystallizer to create fine crystal ammonium sulfate which is then granulated (not shown). Ammonia vapour enters the gas stream during the carbon capture process in R-1. This vapour is removed by passing through a fill pack where a solution of sulphuric acid pumped across the fill pack surfaces. The ammonia reacts with the thin films of sulphuric acid and creates ammonium sulfate. No ammonia vapour leaves the process.

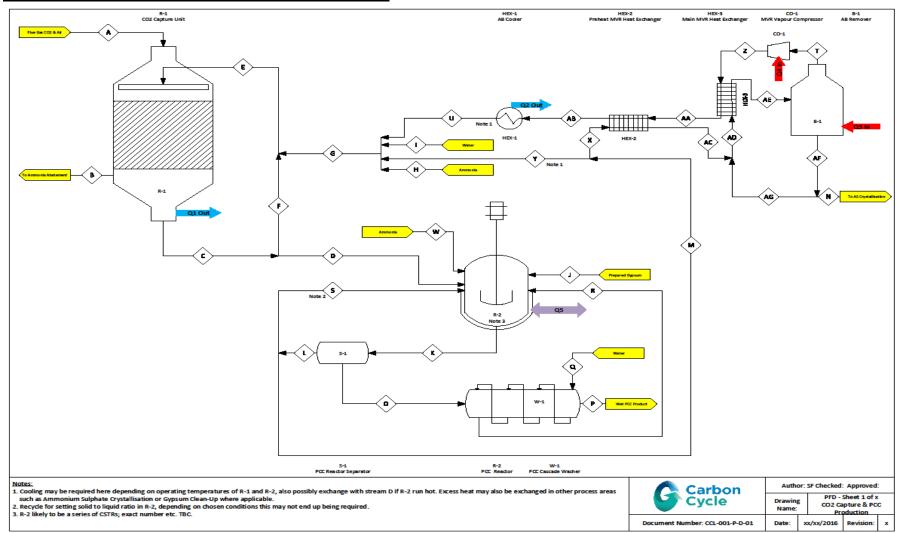


Figure Five: Process Flow Diagram of Carbon Cycle Process

Appendix Four - List of Documents Completed as Part of the FEED Study

The main operational plant areas:

- CCL 1000: Supply and Storage
- CCL 2000: Gypsum Clean-Up
- CCL 3000: Carbon Dioxide Capture and PCC Production
- CCL 4000: Ammonia Scrubbing
- CCL 5000: Water Separation and Ammonium Sulfate heating
- CCL 6000: Ammonium Sulfate Cooling and Ammonia Bicarbonate Removal
- CCL 7000: PCC Washing
- CCL 8000: Ammonium Sulfate Crystallisation
- CCL 9000: Final Product Storage
- CCL 10000: Effluent Treatment and Storage

The following detailed design documents were produced:

Basis of Design - defines the agreed basis to which the FEED Study was to be developed

Process Descriptions – accompanied by process flow diagram, describes the process from the inputs to the outputs

Heat and Material Balance – calculation of the mass and energy balance for the process

Process Control Systems – outlines how the process will be monitored and controlled

Product Descriptions – lists the feed and final products and their physical format

Capacity Analysis – lists the plant input and output values

Equipment Lists - a list of the equipment used in the process

Instrument Lists – a list of the instruments used in the process

Line Lists – a list of the pipe lines used in the process

Valve Lists – a list of the valves used in the process

Equipment Datasheets – provides further information on the equipment used in the process

P&IDs – the piping & instrumentation diagrams, with native files

PFDs – the process flow diagrams

Structural Diagram – shows drawings of the process plant in 3D view

HAZID Study – identifies the hazards in the process, causes, consequences and actions to be taken in order to eliminate or reduce them

Risk Assessment Report – an overall report which includes the HAZID study as well as risk assessment with risk ratings

Appendix Five: List of Prosim Scenarios Examined

- a) 0% ammonium bicarbonate in R-2
- b) 2% ammonium bicarbonate in R-2
- c) 5% ammonium bicarbonate in R-2
- d) 8% ammonium bicarbonate in R-2

A number of other minor cases and variations were also looked at.

Appendix Six: Profit Statement for Alberta Plant

This is for a plant producing 29,120 tons of PCC per year and 60,448 tons of ammonium sulphate per year. Plant operates 320 days per year.

| Period Starts | | | 01/01/2020 | 01/01/2021 | 01/01/2022 | 01/01/2023 | 01/01/2024 | 01/01/2025 | 01/01/2026 | 01/01/2027 | 01/01/2028 | 01/01/2029 | 01/01/2030 |
|-------------------------------------|---------|----------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|
| | | | | | | | | | | | | | |
| Income Statement | Units | Total | | | | | | | | | | | |
| Revenue | | | | | | | | | | | | | |
| Total Revenue | GBP 000 | 79,486 | 3,325 | 6,750 | 6,851 | 6,954 | 7,058 | 7,164 | 7,272 | 7,381 | 7,491 | 7,604 | 7,718 |
| Operating Costs | | | | | | | | | | | | | |
| Total Operating Costs - Commodities | GBP 000 | (22,370) | (936) | (1,900) | (1,928) | (1,957) | (1,986) | (2,016) | (2,047) | (2,077) | (2,108) | (2,140) | (2,172) |
| Total Operating Costs - Labour | GBP 000 | (11,347) | (412) | (958) | (976) | (994) | (1,013) | (1,032) | (1,052) | (1,072) | (1,093) | (1,114) | (1,135) |
| Total Operating Costs - Other | GBP 000 | (12,264) | (383) | (1,028) | (1,052) | (1,075) | (1,100) | (1,125) | (1,151) | (1,177) | | | |
| Total Operating Costs | GBP 000 | (45,981) | (1,731) | (3,886) | (3,955) | (4,027) | (4,099) | (4,173) | (4,249) | (4,326) | (4,405) | (4,486) | (4,568) |
| EBITD | GBP 000 | 33,505 | 1,594 | 2,864 | 2,896 | 2,927 | 2,959 | 2,991 | 3,023 | 3,054 | 3,086 | 3,118 | 3,150 |
| Depreciation | GBP 000 | (9,876) | (494) | (494) | (494) | (494) | (494) | (494) | (494) | (494) | (494) | (494) | (494) |
| Interest Paid | GBP 000 | (3,093) | (295) | (545) | (486) | (427) | (368) | (309) | (250) | (191) | (133) | (74) | (15) |
| EBT | GBP 000 | 20,536 | 806 | 1,826 | 1,916 | 2,006 | 2,097 | 2,188 | 2,278 | 2,369 | 2,460 | 2,551 | 2,642 |
| Tax Payable | GBP 000 | (4,107) | (62) | (266) | (284) | (303) | (321) | (339) | (357) | (375) | (393) | (411) | (627) |
| PAT | GBP 000 | 16,429 | 743 | 1,559 | 1,632 | 1,704 | 1,776 | 1,849 | 1,921 | 1,994 | 2,067 | 2,139 | 2,014 |

| Abbreviations List | |
|--------------------|--|
| CAPEX | Capital Expenditure |
| OPEX | Operating Expenditure |
| EBITD | Earnings before Interest, Tax & Depreciation |
| EBIT | Earnings before Interest & Tax |
| PAT | Profit after Tax |
| GAAP | Generally Accepted Accounting Principles |
| IRR | Internal Rate of Return |
| IFRS | International Financial Reporting Standards |
| NPV | Net Present Value |
| ROE | Return on Equity |
| DSCR | Debt Service Coverage Ratio |
| ISCR | Interest Service Coverage Ratio |
| COD | Commercial Operation Date |

Appendix Seven: Carbon Footprints of Production Plants Operating in Alberta

Two variations were looked at:

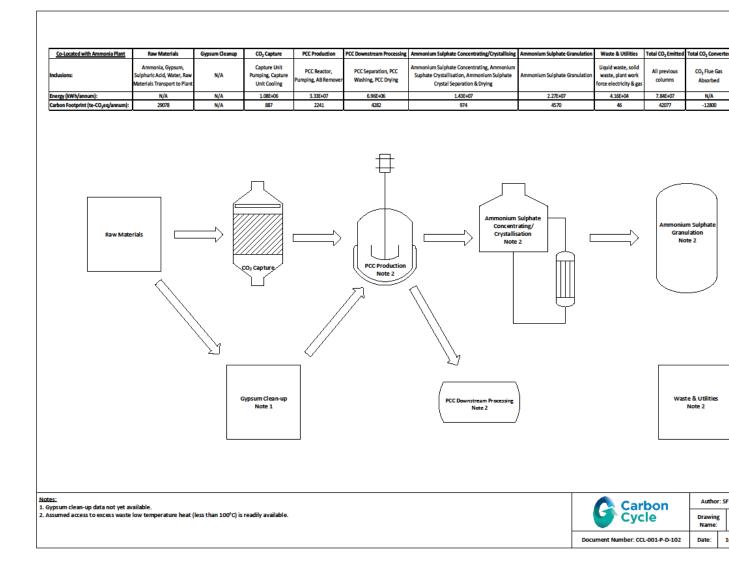
- a) Operation of process at an ammonia plant. Dried PCC is produced.
- **b)** Operation at a paper mill. PCC slurry is produced.

In both scenarios, ammonium sulphate is crystallized as fine crystals and then granulated. Gypsum is delivered to each plant from a working gypsum quarry outside of Alberta. If a quarry was opened in Alberta, the distances and the costs would likely be lower. We have assumed that the gypsum is being imported from Kamloops, BC to either an ammonia plant north of Edmonton or the paper mill in Whitecourt. For the paper mill, we are assuming that the ammonia comes from the plant north of Edmonton.

The comparison carbon footprint figures for production of ammonium sulphate and PCC are published values we found. Looking at the figures, we suspect that the published numbers have been skewed to be lower than they actually are. Actual figures could be higher. We have used the published figures. We do not have data for Alberta. The carbon footprint value for PCC is different that the value that we used in the past. The previous value was given to us by a consultant. As part of preparing this work, we checked his references. He read a table wrong. The correct value is now being used.

These figures reflect no potential gains from further process integration where we transfer waste heat from one part of the process to another and make energy savings. We plan to work on this in the future.

Operation of Process at an Ammonia Plant



Operation of Process at an Ammonia Plant

| | | Volume | | - | Energy/ | | | CO2 Footprint | | - | Total CO2 Footprin |
|----------------------------|---|-------------|--------------------------------|-----|---------|-----------------------|-------|---------------|---------------------------------|-------|--------------------|
| | | /Mass Value | Volume/Mass Unit | Ref | | Energy/ Distance Unit | Ref | Value | CO2 Footprint Unit | Ref | (te-CO2eq/te-CO2 |
| hemical Inputs | | | | - | | | | | | - | |
| Gypsum | | _ | | + | | | + | | | - | |
| - market | Footprint From Mine | 411 | te-gypsum/te-CO2 | 1 | N/A | N/A | N/A | 0.00238 | te-CO2eq/te-gypsum | 45 | 0.0098 |
| | Rail Transport From Mine to Ammonia Plant | 4.11 | te-gypsum/te-CO2 | 1 | 756 | km | 2 | 19 | g-CO2eg/tkm | 67 | 0.0590 |
| Anhydrous Ammonia | | - | the higher of the cost | - | 120 | | - | | h conception | | 0.0350 |
| Annyarous Annona | Producted Design Association | 123 | to 1012 to 002 | 6 | 31.8 | C10- 1012 | 42 | 56.1 | he collection | 42 | 2.2008 |
| | Footprint From Ammonia Supplier | 123 | te-NH3/te-CO2 | 6 | 31.5 | GI/te-NH3 | 60 | 0.820 | kg-CO2eq/GJ | 7 | 0.0000 |
| | Transport From Ammmonia Supplier To CCL Plant | 1.45 | te-NH3/te-CO2 | | U | kWh/te-NH3 | 60 | 0.820 | kg-CO2eq/kWh | | 0.0000 |
| Sulphuric Acid | | | | - | | | | - | | | |
| | Footprint From Sulphuric Acid Supplier | 133 | te-SA/te-CO2 | 21 | N/A | N/A | N/A | 0 | te-CO2eq/te-H2SO4 | 43 | 0.0000 |
| | Transport From Sulphuric Acid Supplier To CCL Plant | 1.35 | te-SA/te-CO2 | 21 | 10 | km | - 22 | 62 | g-CO2eq/tkm | 67,22 | 0.0008 |
| CO2 Flue Gas | | | | _ | | | | | | | |
| | Footprint From Flue Gas Supplier | 33.33 | te-FG/te-CO2 | 8 | N/A | N/A | N/A | -0.03 | te-CO2eq/te-FG | 8 | -1.0000 |
| | Transport From Flue Gas Supplier To CCL Plant | 33.33 | te-FG/te-CO2 | 8 | 0 | kWh/te-FG | 9 | 0.820 | kg-CO2eq/kWh | 7 | 0.0000 |
| Water | | | | | | | | | | | |
| | Footprint From Supply | 3.84 | m ⁸ -H2O/te-CO2 | 23 | N/A | N/A | N/A | 0.344 | kg-CO2eq/m ⁸ -H2O | 10 | 0.0013 |
| aste Outputs | | | | | | | | | | | |
| Solid Waste | | | | | | | | | | | |
| | Gypsum Contaminants (Treat As Sand) Transport To Disposal | 0.10 | te-Cont./te-CO2 | 11 | 10 | km | 24 | 62 | g-CO2eq/tkm | 67,24 | 0.0001 |
| | Gypsum Contaminants Disposal | 0.10 | te-Cont./te-CO2 | 11 | N/A | N/A | N/A | 2.0 | kg-CO2eq/te-Cont. | 25 | 0.0002 |
| Liquid Waste | | | | + | | | - | | | - | |
| | Sewage | 3.84 | m ² -Sewage./te-CO2 | 26 | N/A | N/A | N/A | 0.708 | kg-CO2eq/m ⁴ -sewage | 12 | 0.0027 |
| C Production | Joint Contract of | 2.04 | | | 19/0 | 176 | 19/10 | 0.700 | | | 0.0027 |
| Prosim Simulation | | _ | | - | | | - | | | - | |
| Prosim Simulation | Particles | | 21/2 | | 264.0 | hits &= 603 | - | 0.030 | he consulture | | |
| | Eletricity | N/A | N/A | N/A | 261.8 | kWh/te-CO2 | 13 | 0.820 | kg-CO2eq/kWh | 7 | 0.2147 |
| | Gas | N/A | N/A | N/A | 2386.9 | kWh/te-CO2 | 13 | 0 | kg-CO2eq/kWh(Net) | 66 | 0.0000 |
| | Cooling | 0.0263 | kWh-Input/kWh-Cooling | 53 | 1376.9 | kWh/te-CO2 | 13 | 0.820 | kg-CO2eg/kWh | 54,7 | 0.0297 |
| Washing/Seaparation | | | | _ | | | | | | _ | |
| | PCC Centrifuge Separation & Washing | 2.27 | te-PCC/te-CO2 | 18 | 160.6 | kWh/te-CO2 | 52 | 0.820 | kg-CO2eq/kWh | 7 | 0.1317 |
| Solid Transportation | | | | | | | | | | | |
| | PCC Slurry | 45.48 | te-PCC slurry/te-CO2 | 20 | 19.86 | kWh/te-CO2 | 58 | 0.820 | kg-CO2eq/kWh | 7 | 0.0163 |
| Drying | | | | | | | | | | | |
| | PCC Drying To Powder Heat | 2.27 | te-PCC/te-CO2 | 18 | 60 | kWh/te-PCC | 46 | 0 | kg-COZeq/kWh(Net) | 66 | 0.0000 |
| | PCC Drying To Powder Power | 2.27 | te-PCC/te-CO2 | 18 | 100 | kWh/te-PCC | 46 | 0.820 | kg-CO2eq/kWh | 7 | 0.1865 |
| monium Sulphate Production | | | | | | | | | | | |
| Concentrating | | | | - | | | | | | | |
| | Raising Ammonium Sulphate Concentration | 4.79 | te-AS/te-CO2 | 17 | 16.5 | kWh/te-AS | 41 | 0.2016 | kg-CO2eq/kWh(Net) | 15 | 0.0159 |
| Crystallising | | | | - | | | | | | 1 | |
| | Crystallising Ammonium Sulphate Fine Crystal | 4,79 | te-AS/te-CO2 | 17 | 57 | kWh/te-AS | 40 | 0.2016 | kz-CO2eo/kWh/Net) | 15 | 0.0550 |
| | Drying | 4.79 | te-AS/te-CO2 | 17 | 175.7 | kWh/te-AS | 48 | 0 | kg-CO2eq/kWh(Net) | 66 | 0.0000 |
| | Centrifuging | 4.79 | te-AS/te-CO2 | 17 | 6.4 | kWh/te-CO2 | 47 | 0.820 | kg-CO2eg/kWh | 7 | 0.0052 |
| Granulation | | | | 1 | | | 1 | | | | |
| | Granulating Ammonium Sulphate | 4.79 | te-AS/te-CO2 | 17 | 370 | kWh/te-AS | 68 | 0.20160 | kg-CO2eq/kWh | 15 | 0.3570 |
| her | | | | | 270 | | - | 0.000 | | | 2.3370 |
| | | | | + | | | + | | | | |
| Work Force/Ancillary | | 1 | | 1 | | | 1 | | | 1 | |
| power water etc | | | | - | | | - | | | - | |
| | Work Force Electricity | 12800 | te-CO2/yr | 16 | 9600 | kWh/yr | 14 | 0.820 | kg CO2eq/kWh | 7 | 0.0006 |
| 1 | Work Force Gas | 12800 | te-CO2/yr | 16 | 32000 | kWh/yr | 14 | 0 | kg-CO2eq/kWh(Net) | 66 | 0.0000 |

| Competitors Ammonium Sulphate Emission | 0.58 | kg-CO2eq/kg-AS | 61 |
|---|--------|---------------------|----|
| Average Competitors Ammonium Sulphate Emission | 0.58 | kg-CO2eq/kg-AS | |
| Competitors PCC Emission | 185.00 | kg-CO2eq/te-PCC | 62 |
| | 210.00 | kg-CO2eq/te-PCC | 63 |
| Average Competitors PCC Emission | 197.50 | kg-CO2eq/te-PCC | Т |
| Ammonium Sulphate Produced by CCL Process | 4.79 | te-AS | |
| PCC Produced by CCL Process | 2.27 | te-PCC | |
| Total Competitors Ammonium Sulphate CO2 Footprint | 2.78 | te-CO2eq | Т |
| Total Competitors PCC CO2 Footprint | 0.45 | te-CO2eq | |
| Total CO2 Footprint for Both Products from Current Competitors | | | 1 |
| Processes | 3.23 | te-CO2eq | |
| Total CO2 Footprint for Both Products from CCL Process | 2.29 | te-CO2eq | |
| Reduction of CO2 Footprint for Both Products by CCL Process over | | | Т |
| Current Competitors Processes | 0.94 | te-CO2eq | |
| Current Competitors Processes Carbon Footprint per Tonne of Product | 0.45 | te-CO2ea/te-product | Т |
| CCL Process Carbon Footprint per Tonne of Product | 0.46 | te-CO2eq/te-product | +- |
| Reduction of CO2 Footprint for Both Products by CCL Process over | 0.32 | te-cuzed/te-product | + |
| Current Competitors Processes per Tonne of Product | 0.13 | te-CO2eg/te-product | |

References & Assumptions:

41

1 from stoichiometry 3.91g gypsum to 1g CO2, plus 5% for contaminants and losses assumed

2 For shipping gypsum to ammonia plant: Assume ammonia plant is in Medicine Hat and gypsum production is in Kamloops, BC. Total distance is 756 km rail transport

6 2056.12 kg/h NH3 supply from Carbon Cycle Process 1 H&MB Prosim 2% AB in R-2 Case Simulation Report

7 0.820 kg-CO2eq/kWh for Alberta electricity from https://www.bullfrogpower.com/wp-content/uploads/2015/09/2015_bullfrog_power_electricity_emission_calculator.pdf As per National Inventory Report 1990-2013 Greehouse Gas Sources and Sinks, Environment Canada at http://www.ec.gc.ca/ges-ghg

8 Treated as 3% weight CO2

9 Flue gas blower included in prosim model no further transport required.

10 0.344 kg-CO2eq/m3 H2O for water supply from UK Government GHG Conversion Factors for Company Reporting spreadsheet

11 2.5% of gypsum feed is contaminants, based on analysis between 1 and 3%.

12 0.708 kg-CO2eq/m3 sewage for water treatment from UK Government GHG Conversion Factors for Company Reporting spreadsheet

13 3,350,651 kWh/yr electricity + 30,552,008 kWh/yr gas + 17624794 kWh/yr cooling and capturing 12800 te-CO2/yr from Carbon Cycle Process 1 H&MB Prosim 2% AB in R-2 Case Simulation Report

14

Email from Ian: I can find no numbers for this as it is normally swept up in actual numbers reported for electricity, gas, water etc used. I can't see that this is going to be significant in the grand scheme of things so suggest we add in an allowance for electricity, gas, water etc for staff use of maybe the equivalent of two

residential households as we have 10 staff x 3 shifts. Electricity of 9600 kwh per annum, Gas of 32000 kwh per annum.

15 0.2016 kg-CO2eq/kWh(Net) for natural gas (56kg-CO2/GJ) from http://www.nrcan.gc.ca/energy/efficiency/industry/technical-info/benchmarking/canadian-steel-industry/5193

16 CO2 capture per year is 40*320 = 12800te/yr

17 7977.25 kg/hr AS in AS solution from PCC production to AS production from Carbon Cycle Process 1 H&MB Prosim 2% AB in R-2 Case Simulation Report

18 3790.35 kg/hr PCC from PCC production from Carbon Cycle Process 1 H&MB Prosim 2% AB in R-2 Case Simulation Report

19 20% moisture content in PCC cake

20 5% solid in PCC slurry

21 2220 kg/h pure sulphuric acid for PCC production from Carbon Cycle Process 1 H&MB Prosim 2% AB in R-2 Case Simulation Report

22 Assumed sulphuric acid supplier 10 km away and delivery is by truck

23 Water usage assumed as water use for PCC production from Carbon Cycle Process 1 H&MB Prosim 2% AB in R-2 Case Simulation Report, this should be conservative as most water will come from recovered water from AS crystallisation etc

24 Solid contaminant waste assumed transported 10km by truck to disposal.

25 Solid contaminant disposal treated as soil to landfill using emission factor from UK Government GHG Conversion Factors for Company Reporting spreadsheet

26 Sewage volume assumed equal to the water supply

40 Quote from france evaporation for fine crystal from 49.5% AS solution

41 Concentrating energy from 38.4 to 49.5% AS extrapolated from crystallising energy quote via by 57*(49.5/38.4)-57

42 Energy of 31.8GJ/te-NH3 production using natural gas with emission of 56.1kg-CO2/GJ from Yara_2013_Carbon_footprint-of_AN_Method_of_calculation

43 No emission for sulphuric acid production as made as byproduct from energy production

45 Old reference from Europa site of european commission, Sept 2012

46 Updated after talking with Stephen Armstrong. Reference from Attritor.

47 Updated after talking with Stephen Armstrong. Assumed dewater from 50% slurry to 10% water, mechanical dewatering energy is 41.9 MJ/m3-water from basics in mineral processing

48 Assumed dewater from 10% slurry to 5% water, thermal dewatering energy is 2260 MJ/m3-water from basics in mineral processing

- 52 Updated after talking with Stephen Armstrong. Assumed dewater from slurry to 20% water, mechanical dewatering energy is 41.9 MJ/m3-water from basics in mineral processing, AS solution density is 1195 kg/m3 for approx. 40%, contaminants filtered then washed twice thus used three times
- 53

Updated after talking with Stephen Armstrong. From Heuristics in Chemical Engineering: Compression refrigeration with 100°F (38C) condenser requires these HP/ton is 1.24 (0.9246678 kW/ton) at 20°F(-7C) and ton of refrigeration is the removal of 12,000 Btu/hr of heat (3.517 kW); thus 0.9246678/3.517 = 0.263 kWinput/

kW-Cooling

54 Refrigeration energy supplied as electricity for compressor

58 From Pumps document for 3 times 500 GPM 50 Feet Head Pump with Horse Power of 15 converted to kW

60 Assumed ammonia supplied by pipeline from ammonia plant

61

Fertilizers Europe: Energy efficiency and greenhouse gas emissions in European mineral fertilizer production and use

https://www.google.co.uk/url?sa=t&rct=j&q=&esrc=s&source=web&cd=1&ved=0ahUKEwjajon7317PAhUsJsAKHdr_A34QFggcMAA&url=https%3A%2F%2Fwww.researchgate.net%2Ffile.PostFileLoader.html%3Fid%3D5728857d5b4952f9e43714f1%26assetKey%3DAS%253A357614736166 913%25401462273405605&usg=A

FQjCNFynY9Q1HiTa-cShDJz3R4h7BE5XQ&cad=rja

62 PCC satellite vs merchant process models

63 Tier et al (2005)

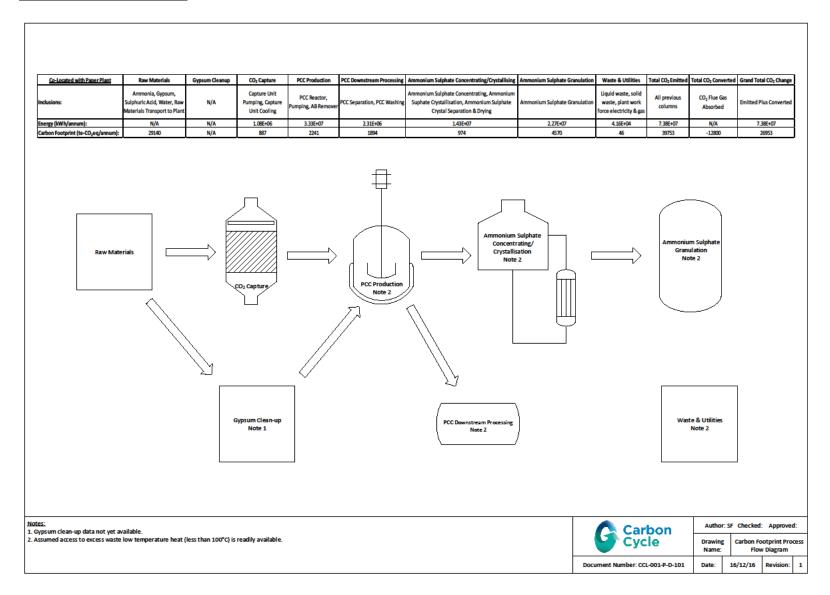
64 Gypsum clean-up process including grinding is not included in the carbon footprint calculation as details are not known yet for this process.

65 Plant life cycle aspects such as plant construction footprint are not included as these values are not publicly available for current processes and our process has not reached sufficient development to produce such detail and so there is no basis for comparison at this time. 66 Assumed access to excess waste low temperature heat (less than 100°C) is readily available.

67 From the Railway Association of Canada: Calculated from the online calculator (100 tons shipped 100 km) Train: 19 g/T/km Truck: 62 g/T/km

68 Fluidised bed granulator assumed and energy assumed as gas as high temp may be needed from http://www.gea.com/en/binaries/GEA Pharma - Granulation-Methods - ART - GBodf tcm11-16923.pdf

Operation at a Paper Mill



Operation at a Paper Mill

| | | Volume | | T 1 | Energy/ | | T | CO2 Footprint | | I | Total CO2 Footprin |
|-----------------------------|---|-------------|--------------------------------|-----|----------------|-----------------------|------|---------------|---------------------------------|-------|--------------------|
| | | /Mass Value | Volume/Mass Unit | Ref | Distance Value | Energy/ Distance Unit | Ref | Value | CO2 Footprint Unit | Ref | (te-CO2eq/te-CO2 |
| hemical Inputs | | - | | | | | - | | | | |
| Gypsum | | | | | | | | | | | |
| | Footprint From Mine | 4.11 | te-gypsum/te-CO2 | 1 | N/A | N/A | N/A | 0.00238 | te-CO2eq/te-gypsum | 45 | 0.0098 |
| | Rail Transport From Mine to Paper Plant | 4.11 | te-gypsum/te-CO2 | 1 | 701 | km | 2 | 19 | g-CO2eq/tkm | 67 | 0.0547 |
| Anhydrous Ammonia | | | | | | | | | | | |
| | Footprint From Ammonia Supplier | 1.23 | te-NH3/te-CO2 | 6 | 31.8 | GJ/te-NH3 | 42 | 56.1 | kg-CO2eq/GJ | 42 | 2.2008 |
| | Transport From Ammmonia Supplier To CCL Plant | 1.23 | te-NH3/te-CO2 | 6 | 205 | km | 60 | 19 | g-CO2eq/tkm | 67 | 0.0048 |
| Sulphuric Acid | | | | | | | | | | | |
| | Footprint From Sulphuric Acid Supplier | 1.33 | te-SA/te-CO2 | 21 | N/A | N/A | N/A | 0 | te-CO2eg/te-H2SO4 | 43 | 0.0000 |
| | Transport From Sulphuric Acid Supplier To CCL Plant | 1.33 | te-SA/te-CO2 | 21 | 205 | km | 22 | 19 | g-CO2eg/tkm | 67,22 | 0.0052 |
| CO2 Flue Gas | | | | | | | | | | - | |
| | Footprint From Flue Gas Supplier | 33.33 | te-FG/te-CO2 | 8 | N/A | N/A | N/A | -0.03 | te-CO2eq/te-FG | 8 | -1.0000 |
| | Transport From Flue Gas Supplier To CCL Plant | 33.33 | te-FG/te-CO2 | 8 | 0 | kWh/te-FG | 9 | 0.820 | kg-CO2eq/kWh | 7 | 0.0000 |
| Water | | | | | | | 1 | | | | |
| | Footprint From Supply | 3.84 | m ⁸ -H2O/te-CO2 | 23 | N/A | N/A | N/A | 0.344 | kg-CO2eq/m ² -H2O | 10 | 0.0013 |
| aste Outputs | | | | | 1910 | | | | | | 0.0015 |
| Solid Waste | | | | | | | - | | | - | |
| 3010 10312 | Gypsum Contaminants (Treat As Sand) Transport To Disposal | 0.10 | te-Cont./te-CO2 | 11 | 10 | km | 24 | 62 | g-CO2eq/tkm | 67.24 | 0.0001 |
| | Gypsum Contaminants Disposal | 0.10 | te-Cont./te-CO2 | 11 | N/A | N/A | N/A | 2.0 | kg-CO2eq/te-Cont. | 25 | 0.0002 |
| Liquid Waste | | 0.20 | te compte cor | | N/O | | 19.0 | 2.0 | ing concepte come | ~ | 0.0002 |
| ciquid waste | - | 3.84 | m ⁸ -Sewage./te-CO2 | 26 | N/A | | N/A | 0.708 | kg-CO2eq/m ⁸ -sewage | 12 | 0.0027 |
| | Sewage | 3.84 | m -sewage./te-coz | 26 | N/A | N/A | N/A | 0.708 | KE-COZEd/m -sewage | 12 | 0.0027 |
| C Production | | | | - | | | - | | | | |
| Prosim Simulation | | | | | | | | | | | |
| | Eletricity | N/A | N/A | N/A | 261.8 | kWh/te-CO2 | 13 | 0.820 | kg-CO2eq/kWh | 7 | 0.2147 |
| | Gas | N/A | N/A | N/A | 2386.9 | kWh/te-CO2 | 13 | 0 | kg-CO2eq/kWh[Net] | 66 | 0.0000 |
| | Cooling | 0.0263 | kWh-Input/kWh-Cooling | 53 | 1376.9 | kWh/te-CO2 | 13 | 0.820 | kg-CO2eq/kWh | 54,7 | 0.0297 |
| Washing/Seaparation | | | | | | | - | | | | |
| | PCC Centrifuge Separation & Washing | 2.27 | te-PCC/te-CO2 | 18 | 160.6 | kWh/te-CO2 | 52 | 0.820 | kg-CO2eq/kWh | 7 | 0.1317 |
| Solid Transportation | | | | | | | | | | | |
| | PCC Slurry | 45.48 | te-PCC slurry/te-CO2 | 20 | 19.86 | kWh/te-CO2 | 58 | 0.820 | kg-CO2eq/kWh | 7 | 0.0163 |
| nmonium Sulphate Production | | | | | | | | | | | |
| Concentrating | | | | | | | | | | | |
| | Raising Ammonium Sulphate Concentration | 4.79 | te-AS/te-CO2 | 17 | 16.5 | kWh/te-AS | 41 | 0.2016 | kg-CO2eq/kWh(Net) | 15 | 0.0159 |
| Crystallising | | | | | | | | | | | |
| | Crystallising Ammonium Sulphate Fine Crystal | 4.79 | te-AS/te-CO2 | 17 | 57 | kWh/te-AS | 40 | 0.2016 | kg-CO2eq/kWh[Net] | 15 | 0.0550 |
| | Drying | 4.79 | te-AS/te-CO2 | 17 | 175.7 | kWh/te-AS | 48 | 0 | kg-CO2eq/kWh[Net] | 66 | 0.0000 |
| | Centrifuging | 4.79 | te-AS/te-CO2 | 17 | 6.4 | kWh/te-CO2 | 47 | 0.820 | kg-CO2eq/kWh | 7 | 0.0052 |
| Granulation | | | | | | | | | | | |
| | Granulating Ammonium Sulphate | 4.79 | te-AS/te-CO2 | 17 | 370 | kWh/te-AS | 68 | 0.20160 | kg-CO2eq/kWh | 15 | 0.3570 |
| her | | | | | | | | | | | |
| Work Force/Ancillary | | | | | | | | | | | |
| power water etc | | | | | | | | | | | |
| r - | Work Force Electricity | 12800 | te-CO2/yr | 16 | 9600 | kWh/yr | 14 | 0.820 | kg-CO2eq/kWh | 7 | 0.0006 |
| 1 | Work Force Gas | 12800 | te-CO2/vr | 16 | 32000 | kWh/yr | 14 | 0 | kg-CO2eq/kWh(Net) | 66 | 0.0000 |

| Competitors Ammonium Sulphate Emission | 0.58 | kg-CO2eq/kg-AS | 61 |
|---|--------|---------------------|----|
| Average Competitors Ammonium Sulphate Emission | 0.58 | kg-CO2eq/kg-AS | |
| Competitors PCC Emission | 185.00 | kg-CO2eq/te-PCC | 62 |
| | 210.00 | kg-CO2eq/te-PCC | 63 |
| Average Competitors PCC Emission | 197.50 | kg-CO2eq/te-PCC | |
| Ammonium Sulphate Produced by CCL Process | 4.79 | te-AS | |
| PCC Produced by CCL Process | 2.27 | te-PCC | |
| Total Competitors Ammonium Sulphate CO2 Footprint | 2.78 | te-CO2eq | |
| Total Competitors PCC CO2 Footprint | 0.45 | te-CO2eq | |
| Total CO2 Footprint for Both Products from Current Competitors | | | |
| Processes | 3.23 | te-CO2eq | |
| Total CO2 Footprint for Both Products from CCL Process | 2.11 | te-CO2eq | |
| Reduction of CO2 Footprint for Both Products by CCL Process over | | | |
| Current Competitors Processes | 1.12 | te-CO2eq | |
| Current Competitors Processes Carbon Footprint per Tonne of Product | 0.46 | te-CO2eq/te-product | |
| CCL Process Carbon Footprint per Tonne of Product | 0.30 | te-CO2eq/te-product | |
| Reduction of CO2 Footprint for Both Products by CCL Process over | | | |
| Current Competitors Processes per Tonne of Product | 0.16 | te-CO2eg/te-product | |

References & Assumptions:

1 from stoichiometry 3.91g gypsum to 1g CO2, plus 5% for contaminants and losses assumed

2 For shipping gypsum to paper plant: Paper plant is located in Whitecourt Gypsum travels from Kamloops, BC : 701 km

6 2056.12 kg/h NH3 supply from Carbon Cycle Process 1 H&MB Prosim 2% AB in R-2 Case Simulation Report

7 0.820 kg-CO2eq/kWh for Alberta electricity from https://www.bullfrogpower.com/wp-content/uploads/2015/09/2015_bullfrog_power_electricity_emission_calculator.pdf As per National Inventory Report 1990-2013 Greehouse Gas Sources and Sinks, Environment Canada at

http://www.ec.gc.ca/ges-ghg 8 Treated as 3% weight CO2

9 Flue gas blower included in prosim model no further transport required.

10 0.344 kg-CO2eq/m3 H2O for water supply from UK Government GHG Conversion Factors for Company Reporting spreadsheet

11 2.5% of gypsum feed is contaminants, based on analysis between 1 and 3%.

12 0.708 kg-CO2eq/m3 sewage for water treatment from UK Government GHG Conversion Factors for Company Reporting spreadsheet

13 3,350,651 kWh/yr electricity + 30,552,008 kWh/yr gas + 17624794 kWh/yr cooling and capturing 12800 te-CO2/yr from Carbon Cycle Process 1 H&MB Prosim 2% AB in R-2 Case Simulation Report

14

Email from lan: I can find no numbers for this as it is normally swept up in actual numbers reported for electricity, gas, water etc used. I can't see that this is going to be significant in the grand scheme of things so suggest we add in an allowance for electricity, gas, water etc for staff use of maybe the equivalent of two

residential households as we have 10 staff x 3 shifts. Electricity of 9600 kwh per annum, Gas of 32000 kwh per annum.

15 0.2016 kg-CO2eq/kWh(Net) for natural gas (56kg-CO2/GJ) from http://www.nrcan.gc.ca/energy/efficiency/industry/technical-info/benchmarking/canadian-steel-industry/5193

16 CO2 capture per year is 40*320 = 12800te/yr

17 7977.25 kg/hr AS in AS solution from PCC production to AS production from Carbon Cycle Process 1 H&MB Prosim 2% AB in R-2 Case Simulation Report

18 3790.35 kg/hr PCC from PCC production from Carbon Cycle Process 1 H&MB Prosim 2% AB in R-2 Case Simulation Report

19 20% moisture content in PCC cake

20 5% solid in PCC slurry

21 2220 kg/h pure sulphuric acid for PCC production from Carbon Cycle Process 1 H&MB Prosim 2% AB in R-2 Case Simulation Report

22 Assumed sulphuric acid supplied same as for NH3 delivery

23 Water usage assumed as water use for PCC production from Carbon Cycle Process 1 H&MB Prosim 2% AB in R-2 Case Simulation Report, this should be conservative as most water will come from recovered water from AS crystallisation etc

24 Solid contaminant waste assumed transported 10km by truck to disposal.

25 Solid contaminant disposal treated as soil to landfill using emission factor from UK Government GHG Conversion Factors for Company Reporting spreadsheet

26 Sewage volume assumed equal to the water supply

40 Quote from france evaporation for fine crystal from 49.5% AS solution

41 Concentrating energy from 38.4 to 49.5% AS extrapolated from crystallising energy quote via by 57*(49.5/38.4)-57

42 Energy of 31.8GJ/te-NH3 production using natural gas with emission of 56.1kg-CO2/GJ from Yara_2013_Carbon_footprint-of_AN_Method_of_calculation

43 No emission for sulphuric acid production as made as byproduct from energy production

45 Old reference from Europa site of european commission, Sept 2012

46 Updated after talking with Stephen Armstrong. Reference from Attritor.

47 Updated after talking with Stephen Armstrong. Assumed dewater from 50% slurry to 10% water, mechanical dewatering energy is 41.9 MJ/m3-water from basics in mineral processing

48 Assumed dewater from 10% slurry to 5% water, thermal dewatering energy is 2260 MJ/m3-water from basics in mineral processing

52 Updated after talking with Stephen Armstrong. Assumed dewater from slurry to 20% water, mechanical dewatering energy is 41.9 MJ/m3-water from basics in mineral processing, AS solution density is 1195 kg/m3 for approx. 40%, contaminants filtered then washed twice thus used three times

53

Updated after talking with Stephen Armstrong. From Heuristics in Chemical Engineering: Compression refrigeration with 100°F (38C) condenser requires these HP/ton is 1.24 (0.9246678 kW/ton) at 20°F(-7C) and ton of refrigeration is the removal of 12,000 Btu/hr of heat (3.517 kW); thus 0.9246678/3.517 = 0.263 kWinput/

kW-Cooling

54 Refrigeration energy supplied as electricity for compressor

58 From Pumps document for 3 times 500 GPM 50 Feet Head Pump with Horse Power of 15 converted to kW

60 For paper mill: Plant is located in Whitecourt Ammonia travels from Fort Saskatchewan 205 km by rail

61

Fertilizers Europe: Energy efficiency and greenhouse gas emissions in European mineral fertilizer production and use

https://www.google.co.uk/url?sa=t&rct=j&q=&esrc=s&source=web&cd=1&ved=0ahUKEwjajon7317PAhUsJsAKHdr_A34QFggcMAA&url=https%3A%2F%2Fwww.researchgate.net%2Ffile.PostFileLoader.html%3Fid%3D5728857d5b4952f9e43714f1%26assetKey%3DAS%253A357614736166 913%25401462273405605&usg=A

FQjCNFynY9Q1HiTa-cShDJz3R4h7BE5XQ&cad=rja

62 PCC satellite vs merchant process models

63 Tier et al (2005)

64 Gypsum clean-up process including grinding is not included in the carbon footprint calculation as details are not known yet for this process.

65 Plant life cycle aspects such as plant construction footprint are not included as these values are not publicly available for current processes and our process has not reached sufficient development to produce such detail and so there is no basis for comparison at this time. 66 Assumed access to excess waste low temperature heat (less than 100°C) is readily available.

67 From the Railway Association of Canada: Calculated from the online calculator (100 tons shipped 100 km) Train: 19 g/T/km Truck: 62 g/T/km

68 Fluidised bed granulator assumed and energy assumed as gas as high temp may be needed fromhttp://www.gea.com/en/binaries/GEA_Pharma_-_Granulation-Methods_-_ART_-_GBpdf_tcm11-16923.pdf

Appendix Eight: Hazid Study

Safety and Operability (HAZID) and Risk Assessment December 2015

Carbon Cycle Limited



Carbon Capture Process FEED Study

02 December 2015

Safety and Operability (HAZID) and Risk Assessment

December 2015

Carbon Cycle Limited

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Executive Summary

1.1 HAZID Study Outcome

The Hazard Identification (HAZID) Study took place in Mott MacDonald Limited's (MML) Altrincham office on the 3rd November 2015.

The objectives of the HAZID Study were to:

- Identify safety and operability related hazards related to the operation and maintenance of the CCL process plant.
- Determine the seriousness of the consequences for the identified hazards;
- Identify existing engineering and procedural safeguards that will reduce the consequences related to the hazards;
- Evaluate the adequacy of existing engineering and procedural safeguards;

A total of 72 actions were raised during the HAZID study. The detailed records of the HAZID are attached as Appendix A.

1.2 Risk Assessment Outcome

The Risk Assessment took place in MML's Altrincham office on the 23rd November 2015.

The objectives of the Risk Assessment Study were to:

- Provide an indicative risk ranking of the hazards;
- Recommend additional safeguards and improvements for further risk reductions where necessary

The risk assessment was then carried out post the HAZID study and where the risk hazards were categorised as the following: -

- 16 Intolerable risk hazards
- 25 Tolerable risk hazards
- 5 Negligible risk hazards
- 35 Negligible risk hazards not evaluated in the risk assessment.

The 16 Intolerable risk hazards will require further detailed analysis in a future Hazard and Operability (HAZOP) Study. These potential hazards categorised as Intolerable risks, stems from the following areas of concern:-

- Environmental Impact
- Process Hazards

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- Accommodation and Non Process Hazards
- Working Environment
- Other Hazards
 - Maintenance
 - Location

The 35 Negligible risk hazards that were not evaluated in the risk assessment were mainly due to the fact that these hazards were heavily dependent on the location of the proposed plant.

332725/PPN/NNP/RP2/P2 02 December 2015 332725/PPN/RP2/P2 For the purpose of the HAZID the plant location was not defined but for the HAZOP and permitting regulations, an assumed location of the plant will required as this will allow these hazards to be evaluated in detail.

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- i.



2 Introduction

2.1 Background

Carbon Cycle Limited (CCL) is developing a new process Carbon Capture Process for making two bulk chemicals - precipitate Calcium Carbonate (PCC), which is used in the paper industry, and Ammonium Sulphate which is used as a fertiliser. The key benefits stated by CCL are: lower energy use compared to other conventional techniques for manufacturing these chemicals, Carbon Dioxide savings through this increased energy efficiency, and the potential for the technology to be used for Carbon Capture and Storage.

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CCL have commissioned Mott MacDonald to conduct a Front End Engineering Design (FEED) Study which incorporates a Hazard Identification (HAZID) study associated to the process and proposed plant design and to provide a risk assessment of the high risk hazards.

2.2 Scope of HAZID Study

The study examined the potential safety and operability issues related to the CCL process and plant. During the course of the studies, forward actions were agreed and assigned to CCL and/or MML as appropriate. This will ensure that the safety and operability of the facilities are improved with the various actions needed to ensure the risks associated with the hazards foreseen in the Hazard Identification (HAZID) study can be mitigated or eradicated.

2.2.1 Systems to be Subject to HAZID

The following systems were the main subject of the HAZID study.

- Natural Hazards
- Environmental Impact
- Process Hazards
- Accommodation and Non Process Hazards
- Working Environment
- Other

The studies also examined the layout of the process plant.

2.3 HAZID Nodes

The following are the nodes that were used for the HAZID studies.

- Natural hazards
 - Extreme Weather
 - Lightning
 - Seismic Activity
- Environmental impact
 - Discharge to Air
 - Discharges to Water
 - Discharges to Vapour

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Safety and Operability (HAZID) and Risk Assessment

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- Discharges to Soil
- Location and Layout
- Process Hazards
 - Process
 - Process Releases Unignited
 - Process Releases Ignited
 - Process Temperatures
 - Process Pressures
 - Services Failure
 - Control Systems Failure
 - Moving Parts Process
 - Materials Handling
 - Sampling
- Accommodation and Non Process Hazards
 - Non Process Fires
 - Smoke Ingress
 - Gas Ingress
 - Working at Height
 - Vehicle Movements
 - Stacking and storage
- Working Environment
 - Physical
 - Temperature
 - Atmospheres
- Other
 - Maintenance
 - Location
 - Security

2.3.1 Documents

The HAZID study used the following drawings and documentation.

| Table 2.1: P&IDs for HAZID | | |
|----------------------------|--------------------------------------|----------|
| Reference | Title | Revision |
| MMD-332725-PID-1000-001 | Carbon Dioxide Supply and Filtration | P1 |
| MMD-332725-PID-1000-002 | Sulphuric Acid Tanker Off Loading | P1 |
| MMD-332725-PID-1000-003 | Sulphuric Acid Storage | P1 |
| MMD-332725-PID-1000-004 | Ammonia Off Loading | P1 |
| MMD-332725-PID-1000-005 | Ammonia Storage | P1 |
| MMD-332725-PID-1000-006 | Aqueous Ammonia Storage | P1 |
| MMD-332725-PID-1000-007 | Gypsum Shipping and Storage | P1 |

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| MMD-332725-PID-1000-008 | Water Supply and Storage | P1 |
|--------------------------|---|----|
| MMD-332725-PID-1000-009 | Ammonia Storage Scrubber | P1 |
| MMD-332725-PID-1000-010 | Aqueous Ammonia Storage | P1 |
| MMD-332725-PID-2000-001 | Gypsum Clean Up Stage 1 | P1 |
| MMD-332725-PID-2000-002 | Gypsum Clean Stage 2 | P1 |
| MMD-332725-PID-2000-003 | Gypsum Clean Up | P1 |
| MMD-332725-PID-3000-001 | CO2 Capture and PCC Production Stage 1 | P1 |
| MMD-332725-PID-3000-001 | CO2 Capture and PCC Production | P1 |
| | Stage 2 | |
| MMD-332725-PID-4000-001 | Ammonia Scrubbing | P1 |
| MMD-332725-PID-5000-001 | Water Separation | P1 |
| MMD-332725-PID-6000-001 | Ammonium Sulphate Cooling | P1 |
| MMD-332725-PID-7000-001 | PCC Washing | P1 |
| MMD-332725-PID-8000-001 | Ammonium Sulphate Production | P1 |
| MMD-332725-PID-8000-002 | Ammonium Sulphate Production | P1 |
| MMD-332725-PID-9000-001 | PCC Drying and Bagging System | P1 |
| | Sheet 1 | |
| MMD-332725-PID-9000-002 | PCC Drying and Bagging System Sheet 2 | P1 |
| MMD-332725-PID-9000-003 | PCC Drying and Bagging System | P1 |
| | Sheet 3 | |
| MMD-332725-PID-9000-004 | PCC Drying and Bagging System | P1 |
| | Sheet 4 | |
| MMD-332725-PID-9000-005 | Ammonium Sulphate Bagging Sheet 1 | P1 |
| MMD-332725-PID-9000-006 | Ammonium Sulphate Bagging Sheet 2 | P1 |
| MMD-332725-PID-10000-001 | Chemical Day Tank for Effluent Storage | P1 |

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The following PFDs were not part of the HAZID scope but were referenced for further information.

| Table 2.2: PFDs & GAs | | |
|-----------------------|------------------------------------|----------|
| Reference | Title | Revision |
| CCL-001-P-D-01 | PFD Drawing Carbon Cycle Process 1 | 4.1 |
| - | MML Plant Layout | DRAFT |

The following other documents were used as background information for the HAZOP.

| Table 2.3: | Other Documentation |
|------------|---------------------|
| Table 2.5. | |

| Reference | Title | Revision |
|----------------|---------------------------|----------|
| CCL-001-P-C-01 | Heat and Material Balance | 4 |

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Carbon Capture Process FEED Study Safety and Operability (HAZID) and Risk Assessment

CCL -001-P-R-04

Process Description

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2.4 Team Composition

The HAZID study was carried out in Mott MacDonald Limited's (MML) Altrincham Office on the 3rd November. The HAZID study team members signed the sign in sheets which can be found in Appendix B.

| Name | Company |
|----------------------|------------------------|
| David Sevier | Carbon Cycle Limited |
| lan Thaxter | Carbon Cycle Limited |
| Stephen Florence | Carbon Cycle Limited |
| Waqas Mirza | Carbon Cycle Limited |
| Saleh Sambwa (CHAIR) | Mott MacDonald Limited |
| Elizabeth Lawrenson | Mott MacDonald Limited |
| Sandy Nimmo | Mott MacDonald Limited |
| Michael Page | Mott MacDonald Limited |
| Stephen Armstrong | Mott MacDonald Limited |
| Ghazala Ali-Ahmad | Mott MacDonald Limited |
| Amber Thompson | Mott MacDonald Limited |
| Ged Forkin | Mott MacDonald Limited |

Table 2.4: Risk Assessment Team Composition

2.5 HAZID RISK Assessment Criteria

The HAZID Risk Assessment was carried out on the 23rd November incorporating the following elements.

- HAZID Study of CCL process and associated upstream/downstream processes
- Performing a Risk Assessment
- Analysis of risks for HAZOP

During this process, none of the identified risks have not been mitigated.

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2.6 Team Composition

For the purpose of the HAZID, MML reviewed the Risk Assessment and CCL in conjunction with MML were invited to comment. These comments have been taken into consideration and included in the HAZID Report.

The MML Risk Assessment Team comprised of the following:

| Name | Company |
|----------------------|------------------------|
| Saleh Sambwa (CHAIR) | Mott MacDonald Limited |
| Sandy Nimmo | Mott MacDonald Limited |
| Michael Page | Mott MacDonald Limited |
| Stephen Armstrong | Mott MacDonald Limited |
| Ghazala Ali-Ahmad | Mott MacDonald Limited |
| Amber Thompson | Mott MacDonald Limited |

Table 2.5: Risk Assessment Team Composition

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3 Methodology of the HAZID

3.1 HAZID Study Objectives

The objectives of the HAZID studies were to:

- Identify safety and operability related hazards related to the operation and maintenance of the CCL process plant;
- Determine the seriousness of the consequences for the identified hazards;
- Identify existing engineering and procedural safeguards that will reduce the consequences related to the hazards;
- Evaluate the adequacy of existing engineering and procedural safeguards;

3.2 Methodology

The HAZID analysis is intended to identify hazards which are not necessarily localised, and which may have a wider sphere of influence. Typically, such hazards can arise from the interaction of a particular system with its external environment. For example, external events such as fire, lightning strike or maintenance activities may impact on the facility to cause operational hazards that may need to be addressed in terms of safety. This HAZID method uses 'keywords' applied to consideration of zones of the facility. This method is quite similar to the Preliminary Hazard Analysis (PHA) technique.

3.3 HAZID Keywords

The deviations used in the HAZID are as shown in table 3.1.

| Table 3.1: HAZID Deviations | |
|-------------------------------|-----------------|
| Potential Hazards | Guide Words |
| Natural Hazards | Extreme Weather |
| | Lightning |

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| | Seismic Activity |
|----------------------|------------------------------|
| Environmental Impact | Discharges to Air |
| | Discharges to Water |
| | Discharges to Vapour |
| | Discharges to Soil |
| | Location and Layout |
| Process Hazards | Process |
| | Process Releases – Unignited |
| | Process Releases - Ignited |
| | Process Temperatures |
| | Process Pressures |
| | |

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Safety and Operability (HAZID) and Risk Assessment

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| <u> </u> | • · · · · · | |
|---------------------------------------|-------------------------|--|
| | Sanvisaa Eailura | |
| | Services Failure | |
| | Control Systems Failure | |
| | Moving Parts - Process | |
| | Materials Handling | |
| | Sampling | |
| Accommodation and Non Process Hazards | Non Process Fires | |
| | Smoke Ingress | |
| | Gas Ingress | |
| | Working at Height | |
| | Vehicle Movements | |
| | Vehicle Movements | |
| | Stacking and Storage | |
| Working Environment | Physical | |
| | Temperature | |
| | Atmosphere | |
| Others | Maintenance | |
| | Location | |
| | Security | |
| | · · · | |

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4 Methodology of the Risk Assessment Ranking

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4.1 Introduction

A risk is the likelihood that a hazard will actually cause its adverse effects, together with a measure of the severity of the effect. In practice, likelihood can be expressed as probabilities (e.g. "one in a thousand"), frequencies (e.g. "1 per 100 years") or in a qualitative way (e.g. "occasional", "improbable", etc.). Th severity of the effect can be described in terms of injury to personnel, damage to plant or harm to the environment.

4.2 Initial Risk Assessment

The hazards identified that appeared to pose a significant risk to personnel, the facilities or the environment underwent a risk ranking procedure based on the following frequency and severity categories.

4.2.1 Frequency

T 1 1 4 4

The frequency category is determined based on the likelihood of a hazard occurring, as in Table 4.1.

| Band | Description | Qualitative Interpretation | Interpreted for System Lifecycle |
|------|-------------|---|----------------------------------|
| 5 | Frequent | The event will be continually experienced. | Daily to monthly |
| 4 | Probable | Will occur several times. The event can be expected to | Monthly to yearly |
| | | occur frequently | |
| 3 | Occasional | Likely to occur several times. The event can be expected to occur several times | Yearly to every 10 years |
| 2 | Remote | Likely to occur sometime in the systems lifecycle. It can reasonably be expected to occur several times. | 10 years to 100 years |
| 1 | Improbable | Unlikely to occur, but possible. It can be assumed the | 100 years to 1000 years |
| | | event is unlikely to occur | |

4.2.2 Severity

The severity category is determined based on the potential impact of a hazard, as in table 4.2.

| Table 4.2: | Risk Ranking - Severity | | | |
|------------|-------------------------|--|-----------------------|---|
| Band | | | Operability | Environmental |
| 1 | Minor | Possible single non- | Unscheduled repair or | Limited damage to |
| | | reporting injury | maintenance | minimal area of low |
| | | | | significance |
| 2 | Moderate | Worker/user single minor injury or multiple non- | Single day Plant Loss | Minor effects on biological or physical environment |

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Carbon Capture Process FEED Study Safety and Operability (HAZID) and Risk Assessment

| Band | Description | O o forta | Operability | Environmental |
|------|--------------|---|--------------------|---|
| | Description | Safety | Interpretatio | Environmental |
| | | reportable injuries | | Minor short-medium |
| | | Trespasser single major injury or multiple minor | | term damage to small area of limited significance |
| | | injuries | | Moderate effects on |
| | Significant | Worker/user single major injury or multiple minor injuries | | biological or physical environment (air, water) but not affecting ecosystem |
| 3 | | Trespasser single | ween | function |
| | | fatality, or between two and ten major injuries | | Moderate short- medium term widespread impacts (e.g. significant spills) |
| | | | | Serious environmental effects with some impairment of ecosystem function |
| | - | | | Relatively widespread medium-long term impacts |
| | Catastrophic | Worker/user multiple | Weeks or months of | Very serious environmental effects |
| | | fatalities | plant outage | with impairment of ecosystem function |
| 4 | | | | Long term, widespread effect on significant environment (e.g. wet areas, tidal zones, mangroves) |

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4.2.3 Risk Assessment Matrix

The following matrix was used for the initial assessment of risks at the HAZID.

Table 4.3:Risk Assessment MatrixRisk Table

4.2.4 Calculating Risk Rating

Risk rating score is calculated by multiplying the severity (S) band by the frequency (f) band.

| Table 4.4: Risk Rating Significance | |
|-------------------------------------|---|
| Score | Significance |
| <8 | Negligible - no action required |
| 8-14 | Tolerable risk – Further Assessment may be required |
| 15+ | Intolerable Risk – Assessment required |

| | | | | Severit | y Band | | |
|---------|------------------|--------------|-------------|-------------|------------|------------|------------------|
| | | 5 | 4 | 3 | 2 | 1 | 0 |
| Frequen | cy Band | Catastrophic | Critical | Significant | Moderate | Minor | Not evaluated |
| 5 | Frequent | Intolerable | Intolerable | Intolerable | Tolerable | Negligible | - |
| 4 | Probable | Intolerable | Intolerable | Tolerable | Tolerable | Negligible | - |
| 3 | Occasional | Intolerable | Tolerable | Tolerable | Negligible | Negligible | - |
| 2 | Remote | Tolerable | Tolerable | Negligible | Negligible | Negligible | - |
| 1 | Improbable | Negligible | Negligible | Negligible | Negligible | Negligible | - |
| 0 | Not evaluated | - | - | - | - | - | - |

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5 HAZID and Risk Assessment Records and Findings

5.1 Summary of HAZID Records

The risk assessment included an initial estimation of the risks from the hazards as an aid to selecting hazards for further analysis. A detailed HAZOP study is recommended to be carried out on the hazards identified in the Risk Assessment as Intolerable.

| | | | | Risk Rating | | | |
|--|------------|---------------|---------|-------------|-----------|-------------|--|
| Meeting | Node No | No of Records | Actions | Negligible | Tolerable | Intolerable | |
| Natural Hazards | 1 | 8 | 11 | 1 | 6 | 1 | |
| Environmental Impact | 2 | 10 | 9 | 2 | 4 | 3 | |
| Process Hazards | 3 | 43 | 43 | 2 | 13 | 12 | |
| Accommodation and Non Process Hazards | 4 | 10 | 4 | - | 1 | - | |
| Working Environment | 5 | 7 | - | - | - | - | |
| Other | 6 | 3 | 5 | - | 1 | - | |
| Total | | 81 | 72 | 5 | 25 | 16 | |

Table 5.1: Summary of HAZID records

5.2 Risk Assessment Results

The risk assessment was then carried out after the completion of the HAZID study. In total 81 records were made from the HAZID study which included 72 actions, Based on the frequency and severity categories allocated at the Risk Assessment, the following categorisation of the hazards have be summarized:

- 16 Intolerable Hazards
- 25 Tolerable Risk Hazards
- 5 negligible Risk Hazards

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• 35 negligible Risk Hazards not evaluated in the Risk Assessment.

A HAZID Check List and Risk Assessment is attacked to this document Appendix A.

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6 Discussion of HAZID and Risk Assessment Results

6.1 HAZID General Findings

The HAZID study identified the hazards and significant operability issues related to the design and operation of the CCL process. The general findings are detailed below.

6.1.1 Storage Conditions and Handling of Hazardous Materials

The CCL process requires storage of significant amounts of hazardous material most notably sulphuric acid and ammonia. This will need to be considered within the choice of materials within the design. However areas where cross contamination of these hazardous materials could occur will need to be avoided in future further designs of the CCL process and reflected in general arrangement drawings and drainage system schematics for example. This is because sulphuric acid could be involved in dangerous runaway reactions and ammonia streams could produce toxic fumes in various reactions, both of which could cause harm to personnel.

The current P&IDs show the final destination for hazardous materials effluent and drainage routes. The drainage lines are piped to a chemical day tank ready for neutralisation and disposal, offsite in a safe manner. Future general arrangements and drainage systems shall be designed in such a way where the isolation of ammonia and sulphuric acid drainage streams are independent to avoid explosive reactions. Similar preventive measures shall be made for the water and sulphuric acid.

The corrosive nature of sulphuric acid means that materials with which it comes into contact are able to withstand its corrosive and reactive nature. Therefore metals generally would not be the choice of material for handling sulphuric acid in the process. This is due to the fact that sulphuric acid reacts with most metals to produce hydrogen gas which poses an explosive risk to the plant.

In order to minimise risk of storing hazardous materials, it is best practice to reduce the requirement for storage of such substances. Anhydrous and aqueous ammonia solutions are currently proposed to be stored and used onsite. The application of the COMAH regulations to the presence of these materials will depend on quantities stored.

The CCL process has been designed to capture carbon dioxide as a key aspect of the CCL intellectual property for the process. The carbon dioxide used is of a significant quantity and the risks associated with that have been taken into consideration and incorporated in the design.

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During the process design assessment of the carbon dioxide with respect to personnal and the effects of asphyxiation, requirement for high carbon dioxide detection and high carbon dioxide alarms systems have been incorporated in the design.

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6.1.2 Process Control

Temperature control is essential to avoid runaway reactions involving sulphuric acid in the gypsum cleanup process. Temperature monitoring and control of all major vessels have already been considered in the design. Sulphuric acid at excessively low temperatures may freeze which will can adversely impact the operability of the plant.

The CCL process is currently designed to monitor pressure within all major equipment. Pressure control is also included to areas of the plant where the pressure of the equipment is critical to the plants operability and safety. To ensure high quality of product, overall pH control across the process is vital. However inadequate pH control has not been identified as a significant safety issue. The crystalliser unit and capture unit have been identified to be heavily pH dependent processes; therefore HAZID actions have been put in place to ensure this has been addressed in the design.

During detailed design of the process MML has identified that there may be a requirement for manual sampling. As future HAZOP requirement this needs to be risk assessed to mitigate and risk to personnel during the sampling procedures.

Fire and smoke detection will be required throughout the plant. Majority of the firefighting system will be subject to water based hydrants, hoses and sprinklers.

For the area with large storage of sulphuric acid, water based firefighting systems carry a higher risk of explosions therefore appropriate firefighting systems will be a foam based system.

For the area with large storage of ammonia, water based firefighting systems can be used but alternative such as foam based firefighting system application and suitability will be considered.

The CCL process plant location is yet to be determined. For the purpose of the feed study development Carbon Cycle ERA Grand Challenge Round One Final Report Confidential and any future detail design the emergency evacuation routes and emergency procedures will be outlined.

6.1.3 Plant location

The plant location is yet to be confirmed by CCL. Knowledge of location of the site would allow a deeper analysis of the vulnerability to site specific hazards such as common local services failure and operational upsets from the collocated plant.

6.2 Actions Specifically Associated with High Risk Hazards

During the HAZID study, the hazards that had the highest potential to negatively impact personnel, the environmental or equipment were given a high risk rating. When the safeguards detailed in the HAZID were deemed inadequate to combat the high risk hazards, actions were formulated by the HAZID team members to consider further mitigation. These actions are listed below and are further detailed in the HAZID Minutes and Action Sheets (see Appendices A and B).



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 Table 6.1:
 Intolerable Hazards

| _in ≆ No | | | | | |
|----------------|-------------------------|------------------------|----------------------|--|--|
| 1 | Natural | Extreme Weather | Extreme Temperatures | Too hot: The storage of ammonia and sulphuric | Provide adequate cooling of the areas |
| | | | | acid will vent more of its material at higher temperatures | carrying significant amounts of ammonia and sulphuric acid against the extreme design basis/temperature within the building service design. An adequate air conditioning system should be in place for the personnel in the occupied buildings (e.g. control room and admin buildings) |
| | Environmental Impact | Discharges to Air | Fugitive emissions. | See venting (line 9) | See venting (line 9) |
| | Environmental Impact | Discharges to Water | Drainage | contamination of water, nitrogen pollution, eutrophication, fine, plant shutdown, damage to reputation | Design for emission containment in failure modes. Conduct risk assessment |
| | Environmental Impact | Discharges to Water | Reactions | Increased operating cost, fines, reputational damage, plant shutdown, further treatment required of | Conduct risk assessment. Plan adequate spill/leak detection and treatment for site. |
| | | | | leaked/spilled material, | |
| 0 | | | | increased corrosion | |
| | | | | rates | |

| Process Hazards | Process | Reactions. | Heat, equipment damage. | Ensure adequate pH control, safe heat |
|-----------------|---------|----------------------|---|--|
| | | | | exchange, adequate distances between hazardous material storage areas |
| Process Hazards | Process | Reactions. | Heat, equipment damage. | |
| Process Hazards | Process | Reactions | Vacuum effect and associated damage to tanks | |
| Process Hazards | Process | Reactions. | Explosion. | Prevent metal reaction with sulphuric acid |
| Process Hazards | Process | Corrosive materials. | Hydrofluoric acid formation during gypsum treatment, HF | 1. Consider Teflon-lined vessels/pipes/tubes, give consideration to |

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| | | | | is a major health risk to personnel | personnel protection, such as gas detection. 2. Conduct experiments to determine the extent of reaction to form hydro fluorides. |
|------|-------------------|----------------------------------|---------------------|---|---|
| | Process Hazards | Process | Materials Delivery. | Cost due to loss of raw materials, increased health risk, risk to offloading personnel | Include dosing skid beside flotation tanks, consider storage of flocculants and polymer |
| 26 | | | | | additive |
| | Process Hazards | Process | Gas clouds. | Death of personnel, | Include gas detection |
| | | Releases - Unignited | | local environmental damage, required | system and evacuation to designated safety |
| | | | | report to EA, fines, reputation damage. | areas. |
| 27 | Process Hazards | Process | Asphyxiation. | Death to personnel, | Include detection |
| | | Releases - Unignited | | plant shutdown, report to local authorities and | systems for all Asphyxiate gases, |
| | | | | EA. | procedures for safe isolation of plant and man access to confined spaces. |
| 29 | Process Hazards | Process Releases - Ignited | Fire | Equipment damage, fugitive emissions, plant shutdown, health risk to personnel, reputation damage, legal fees and environmental fines | Include inherent safety and best practices within the design and layout of the site. Include adequate fire and heat detection |
| | Process Hazards | Process | Explosion | Overpressure, death to | Include inherent safety |
| | | Releases - | | personnel, equipment | and best practices within |
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| | Ignited | | damage, plant shutdown, reputation damage, legal fees and environmental fines, subsequent explosions | the design and layout of the site. |
|-----------------|-----------------------|---------|--|------------------------------------|
| Process Hazards | Process | Smoke | Asphyxiation of | Include smoke detection |
| | Releases - Ignited | | personnel, plant shutdown. | |
| Process Hazards | Process | Others. | Collapse/failure of | |
| | Pressures | | equipment | |

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7 Conclusions

7.1 Conclusions

The HAZID study successfully achieved its goal to identify the hazards associated with the operability relating to the CCL process plant. The HAZID covered all areas of the feed study design,, however, due to changes to the CCL process, some areas of MML feed study design were deemed redundant and removed and updated in accordance with the CCL requirements. In circumstances where documentation was limited discussion were had with CCL to establish a way forward and subsequently these will be included in the final feed study report.

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In total 81 records were made from the HAZID study. Hazards that posed a significant risk were allocated into 'frequency' and 'severity' categories and thus into high, medium and low risk ratings during the HAZID study. This has provided a basis for identifying which hazards should be subject to detailed risk assessment. A total of 72 actions were raised at the HAZID. 16 of the actions were associated to hazards with high risk ratings (and therefore should be considered further in a future HAZOP study), 25 were associated to hazards with medium risk ratings and 5 were associated to hazards with low risk ratings. The remaining 35 actions were raised against hazards that were not given a risk rating.

7.2 Recommendations

The HAZID checklist and HAZID risk Assessments are live documents and for the purposes for the feed study is the responsibility of CCL. It is the responsibility of CCL to continuously update these documents throughout the live cycle of this project. Any identified outstanding actions should be completed in a timely manner throughout the life cycle of the project.

7.2.1 Procedures

The following procedures have been identified and should be carried out in accordance with CCL procedures.

A future HAZOP should take into consideration the HAZID Checklist and Risk Assessment produced during the feed study and this should be reviewed in accordance with the CCL Safety and Operability

- Procedures. MML would welcome the opportunity to facilitate the safety management of the project The future HAZOP study should revisit the 16 (intolerable) actions which were associated with high risk rating and any other actions that were identified as tolerable that may need further assessment The Basis of Design, Process Description, Mass Balance, Process Flow Diagrams and P&IDs should be completed and frazer before conducting a future HAZOP.
- be completed and frozen before conducting a future HAZOP

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7.2.2 Highlighted HAZID Study Outcome

 The Heat Balance needs verifying to ensure the amount of heat required to the process could be determined

A full analysis of the impurities in the raw materials will need to be confirmed. e.g fluorites in gypsum

- During the HAZID study the potential of having fluorite impurities within the raw gypsum supply was identified and further action will need to be taken to either remove the fluorite impurity on site safely or
- a supplier of gypsum without this impurity will be preferred. This is because the fluorite impurity may react within the CCL process to produce hydrogen fluoride / hydrochloric acid which will cause major implications equipment and material selection and the operability and safety of the process plant. CCL and MML need to specify the inventory of potentially hazardous materials to be stored on the site, as there may be implications to planning permissions and reporting to other relevant authorities. Consider alternative extinguishants (e.g. foam based) in areas where water based fire fighting systems will cause a high risk hazard for example sulphuric acid Storage area
- Upon confirmation of the proposed collocated plant, a process upset analysis should be carried out to investigate the implications to the CCL process caused by the interface with the collocated plant. e.g loss of pressure of carbon dioxide supply/ammonia.

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8 References

1 1. The Control of Major Accident Hazards Regulations (Third Edition) 2015, Health and Safety Executive