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Section 3. TECHNICAL REPORT

Section 3.1 BACKGROUND

As a primary manufacturing site, [REDACTED] uses a large amount of various solvents in the manufacture of the Active Pharmaceutical Ingredients (API) made on site. As the price of solvents increases, and the environmental legislation surrounding their disposal becomes more stringent, it becomes more and more favourable to recover as much solvent as possible for re-use in the manufacturing processes. One of the API manufacturing processes uses a combination of DiChloroMethane (DCM), DiMethylFormamide (DMF), and Di-IsoPropylEther (IPE). DCM and DMF are both very tightly controlled in terms of how much of each can be legally emitted to the atmosphere, and both have a very high cost of purchase for fresh solvent. As a result of these factors the decision was taken to install a recovery unit onsite whose purpose would be to split the incoming solvent stream into two products, one being a combined DCM/IPE stream (which would be sent to another column for further separation), and the other being a pure DMF product stream.

Section 3.1.1 PROCESS REQUIREMENTS

The process is required to process a feedstock (hereafter referred to as “Crude”), with the composition detailed below: -

Component	% v/v
IPE (Di-IsoPropylEther)	59
DMF (DiMethylFormamide)	29
DCM (DiChloroMethane)	7
H ₂ O (Water)	2
EtOH (Ethanol)	0
Air	2
Salts & Solids	1
Total	100.00

Table 3.1: Feedstock Composition for Processing

If the manufacturing processes which generate this crude are being run to maximum capacity the total volume of crude being produced annually is approximately 5,760,000 l (based on a batch size of 6400 l, with 18 batches per week, and a running time of 50 weeks per year). When this crude is analysed in a cost basis, it can be shown to have a large capital value to the site, as if not recovered, the site would need to buy fresh solvent, which would incur the costs detailed below: -

Component	Cost (£/te)	Mass te/yr	Net Cost (£)
IPE (Di-IsoPropylEther)	995.00	2472	2,459,409
DMF (DiMethylFormamide)	658.00	1625	1,069,204
DCM (DiChloroMethane)	352.00	528	185,835
H ₂ O (Water)	0.00 ¹	114	0
EtOH (Ethanol)	440.00	16	7070
Air	0.00 ²	0	0
Salts & Solids	0.00 ³	51	0
Total		4806	3,721,518

Table 3.2: Crude Cost Breakdown⁴

Table 3.2 shows that the cost of fresh solvent for the site would be approximately £3.7M. So clearly there is a financial driver to recover as much solvent as possible.

Another driver for recovering solvent is that the solvents have high levels of costs associated with them for their disposal, since many of the solvents are tightly controlled under current Environment Agency regulations.

The required product specification for the final DMF product is shown below: -

Component	% v/v
IPE	≤1.00
DMF	≥ 98.70
DCM	≤ 0.1
H ₂ O	≤ 0.2
Total	100.00

Table 3.3: Required Product Specification

Other criteria that require evaluation are:

- ✚ Amount of IPE/DCM product to be recovered.
- ✚ Amount of DMF to be recovered.
- ✚ Amount of Residues left after the batch is complete.

Section 3.1.1.1 IPE/DCM PRODUCT VOLUME

In order to calculate this volume, certain assumptions are required, these being: -

- ✚ Recovery efficiency of IPE circuit.
- ✚ Recovery efficiency of DCM circuit.

Therefore the unit will be designed to recover 90% of all the IPE input into the system, along with 95% of all DCM. This means that the IPE product stream will contain (based on a 28430 l batch size): -

- ✚ 15050 l of IPE
- ✚ 1853 l of DCM

Therefore, ignoring impurities that will be present in the stream, the volume of product recovered will be approximately 16900 l.

Section 3.1.1.2 DMF PRODUCT VOLUME

In order to calculate this volume, certain assumptions are required, these being: -

- ✚ Recovery efficiency of DMF circuit.

Therefore the unit will be designed to recover 80% of all the DMF input into the system. This means that the DMF product stream will contain (based on a 28430 l batch size): -

- ✚ 6712 l of DMF

Therefore, ignoring impurities that will be present in the stream, the volume of product recovered will be approximately 6710 l.

Section 3.1.1.3 RESIDUES

From work performed in lab experiments it was shown that if the concentration of the residues exceeded 56x (in terms of chloride levels) they have a tendency to solidify, which will lead to issues in terms of offloading. Therefore sizing the residues level is important to ensure ease of running and maintenance.

_____ contained details of these residues, detailing the concentrations in the feed being 300 ppmv of chloride. Therefore the limit of the residues size can be calculated: -

$$\text{Residues Volume (l)} = \frac{\left(\frac{\text{Chloride Levels in feed (ppmv)} \times \text{Batch Size (l)}}{\text{Conc of Residues}} \right)}{\text{Chloride Levels in feed (ppmv)}}$$

$$\text{Residues Volume (l)} = \frac{\left(\frac{300 \times 28430}{56} \right)}{300} = 514\text{l}$$

Therefore the decision was taken to restrict the residues volume to no less than 600 l per batch. This result in a chloride concentration of: -

$$\text{Residues Conc.} = \left(\frac{300 \times 28430}{600} \right) = \frac{14215}{300} = 47\text{x which is an acceptable concentration.}$$

Section 3.2 PROJECT STAGES INVOLVED

There were several stages that were required to be completed before the final system design could be confirmed. These stages included: -

1. Project initiation.	PHASE 1
2. User Requirement Brief (URB) review.	
3. Project scope generation.	
4. Review of the existing process and process plant.	
5. Safety screening review.	
6. Materials of Construction review	PHASE 2
7. P&ID design.	
8. P&ID review.	
9. Equipment schedule.	PHASE 3
10. Process plant specification.	
11. Detailed equipment design.	
12. Services schedule.	
13. Draft Control Philosophy.	PHASE 4
14. HAZOP.	
15. SIL assessment.	
16. Software flowchart review.	PHASE 5
17. Procurement.	
18. Construction.	
19. Commissioning.	
20. Handover.	

Section 3.2.1 PHASE 1

Stages involved: -

1. Project initiation.
2. User Requirement Brief (URB) review.
3. Project scope generation.
4. Review of the existing process and process plant.
5. Safety screening review.

Section 3.2.1.1 INITIAL STAGES – PROJECT INITIATION, URB REVIEW, & SCOPE GENERATION

The initial stages of this design were focused around project initialisation and current process equipment and procedure review. The main outcome of this phase of the project was the “Project Scope”, which stated that the process should be re-located to a new location on the site, and that the process be brought into line with current legislation and company guidelines.

Section 3.2.1.2 REVIEW OF EXISTING PLANT

The original process was installed on site in the early 1980’s, and has successfully been in continual use since then. However a recent site rationalisation plan, combined with the unit’s age and expected service lifetime, has forced the site to consider replacement and relocation of the process. The existing process consists of the following setup, which is shown below -

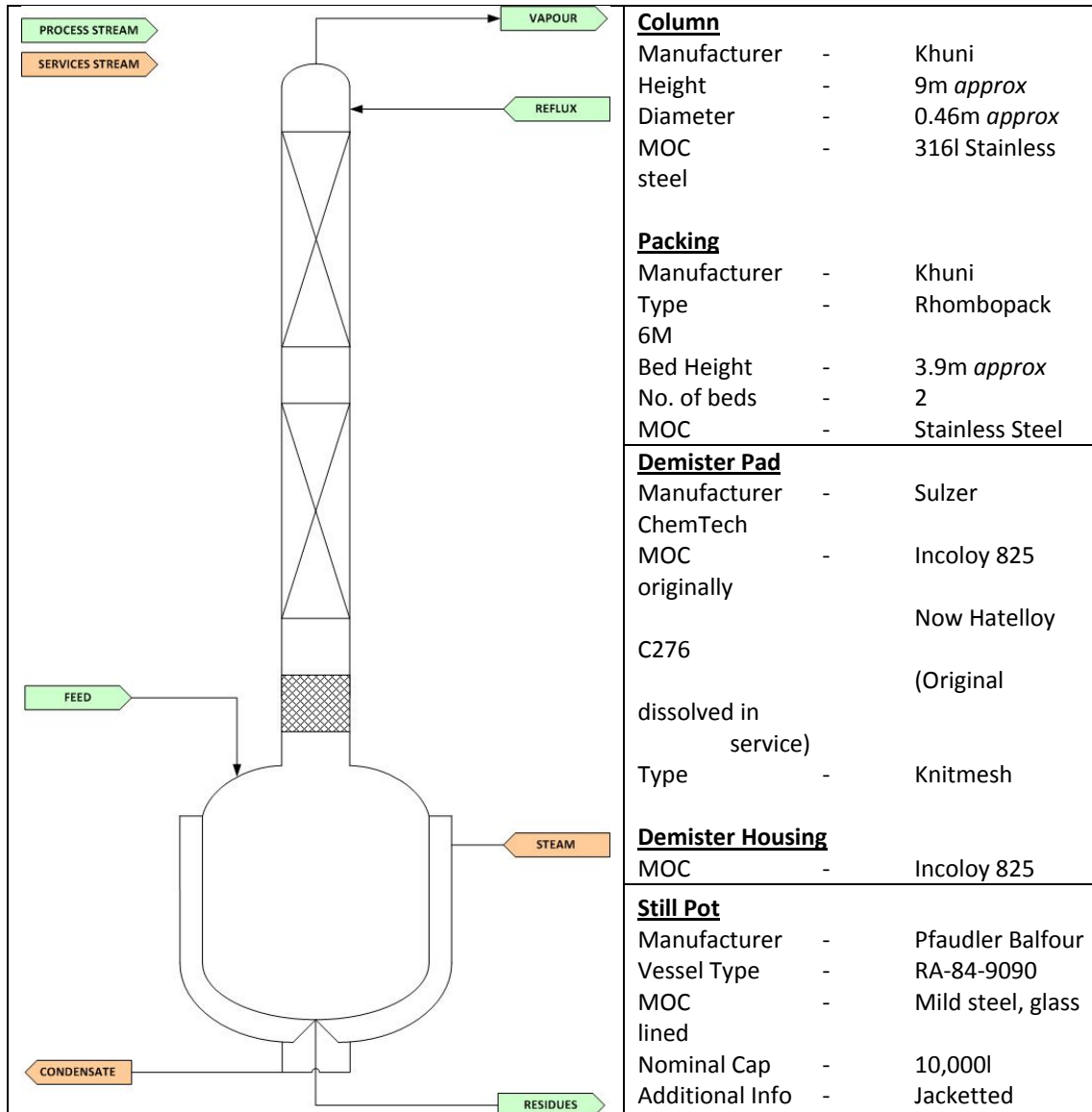


Figure 3.1: Simplified Column & Still Pot Diagram⁵

The column and still pot are the main plant items associated with this process, but in addition to these items are several other associated plant items. A simplified view of the process can be seen overleaf: -

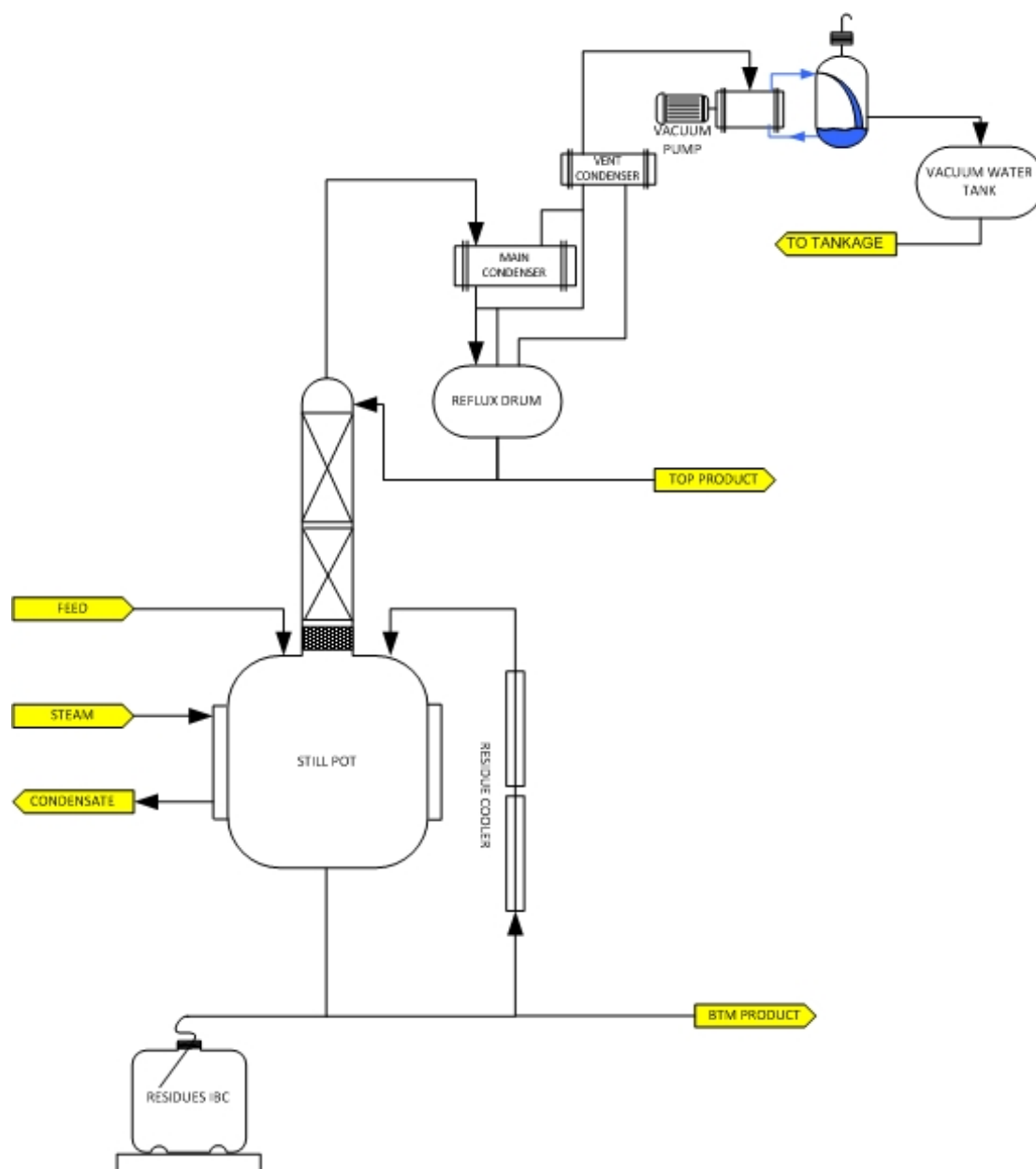


Figure 3.2: Process Overview

Section 3.2.1.3 REVIEW OF EXISTING PROCESS

The original process followed the following simplified process sequence: -

1. Charge the still pot with 6000l of crude.
2. Apply steam to the jacket, and establish a boil-up of 3600l/hr under total reflux.
3. When boil-up is established, reduced reflux ratio to 0.4:1, and introduce crude at such a rate so as to maintain boil-up at 3600l/hr, and to ensure that the still pot level doesn't exceed 8000l.
4. When the still pot temperature reaches 95°C increase reflux ratio to 2:1.
5. When the still pot temperature reaches 115°C, crude to the still pot is ceased.
6. When the still pot temperature reaches 125°C, product is diverted back to crude tanks. Vacuum is applied so that the system pressure is -0.74barg.
7. When the vapour temperature exceeds 104°C, reduce the reflux ratio to 0 (i.e. total products take off).

8. When still pot level until indicated level is 600l. At this point cease steam injection to the jacket.
9. Run the residues through the residue cooler until the temperature is 40°C.
10. Offload residues manually into IBC for disposal.

During the review process, and the subsequent safety screening review (a process designed to identify and where possible design out all safety issues concerned with the plant and process) it was noted that several actions within the operation of the column were undesirable, in particular the offloading of residues from the column. The residue material is an unpleasant material with a tar like consistency if left to cool too much. It also has a highly corrosive nature, as shown not only by laboratory tests done on site, but also by the residues dissolution of a pump impellor during service. The decision was therefore take to review the offload process so as to reduce or remove operator contact during the activity. This issue had also been noted at the last HAZOP carried out on the process, which was reviewed as part of these initial stages.

Having completed these design reviews, initial P&ID's (henceforth referred to as ELD's) were constructed for review. Initially they mimicked those of the original process. However during these initial reviews several key alterations were made, these being: -

- ✚ Position of the IBC is relocated to directly underneath the still pot, so as to allow gravity to empty still pot contents into the IBC. This operation is still to be done manually, but control panel for the process is to be located at a safe distance from the still pot.

Section 3.2.2 PHASE 2

Stages involved: -

1. Materials of Construction review
2. P&ID (ELD) design.
3. P&ID (ELD) review.

Section 3.2.2.1 MATERIALS OF CONSTRUCTION REVIEW

In order to ensure that the column and associated plant items were designed using materials that were fit for purpose, a series of "materials of construction" (MOC) sessions were held, in conjunction with investigations into the effects of the materials used within the process.

On the original plant, the major areas for concern had been: -

1. All residues contact equipment.
2. Demister pad and base sections of packing.

Residue Contact Equipment

Due to the high levels of chlorides that were found to be present in the crude stream (approximately 2% v/v) several alloys become unsuitable for use with residue contact equipment, as detailed overleaf: -

Alloy	Corrosion Rate 2% HCl @B.Pt (mm/yr)	Corrosion Rate 2% HCl plus Fe ³⁺ ion @B.Pt (mm/yr)	Comments
Incoloy 825	~ 2.5		
Hastelloy C-276	~1	<0.5 @ 500 ppm	
Hastelloy C-2000	<0.5		Superior to C-276 in tests. High Cr & 1.6% Cu Resistant to Ferric ion (ref Schwietzer, Encyclopedia of Corrosion Technology)
Hastelloy B-2	<0.13	1.27 @ 50ppm Fe	OK @ <10ppm Ferric
Hastelloy C-4	1.3		
Hastelloy C-22	1.3		
Zirconium	<0.5		10x cost of nickel
Inconel 686	>1.3	<0.13 @500ppm	Low iron form of C-276

Table 3.4: Corrosion Data for Various Alloys when subjected to Residues Conditions

Therefore it can be seen that ideally all parts coming into contact with the residues should be made out of Zirconium. However, due to the high cost of Zirconium, this is not economically viable, therefore it was recommended that these contact parts be made from Hastelloy C-2000, or if that was unavailable, then Hastelloy C-276.

Demister Section

As with the residues, there is a potential issue for chloride and ferric ion levels to cause damage to these parts. Under normal operation this should not be the case, as the vapour generated from the still pot will not contain these components [REDACTED], therefore should not corrode most alloys. However, during recent service (previous 2 years) the unit has “destroyed” two demister pads. This is thought to be down to poor level control on the original equipment which leads to liquid passing into the demister and beyond. Therefore as an added level of protection against process interruptions, the decision was taken to construct the demister housing and pad out of Hastelloy C-276.

Base Sections of Packing

The packing in the original column was stainless steel structured packing. As with the demister pad, it was noticed that after 2 years service the column had lost 2 cakes of packing on one side of the column, this corresponded to where the steam nozzle on the jacket was located. Therefore the conclusion was liquid was passing into the packing, and literally destroying the packing. In order to alleviate this issue, the decision was taken to install a large disentrainment section between the still pot and the column. This would allow for liquid to leave the vapour before entering the demister and crucially before entering the packing. This would then allow the packing and associated column internals to continue to be fabricated from stainless steel. In order to achieve continuity, this disentrainment piece was constructed out of glass line mild steel (i.e. the same materials as the still pot).

Other Points of Note

A known phenomena of stainless steel is a problem with stress corrosion cracking in the presence of low levels of chlorides at temperatures exceeding 50 degC. The site is located in a marine environment, and with operating temperatures routinely exceeding 50 degC, the low levels of chlorides in the local atmosphere have been observed to cause stress corrosion cracks on a number of columns on site. To this end, the decision was taken to fabricate the new column out of Duplex 2205, due to its superior ability to resist attack by stress corrosion.

A simplified view of MOC's to be used can be seen below: -

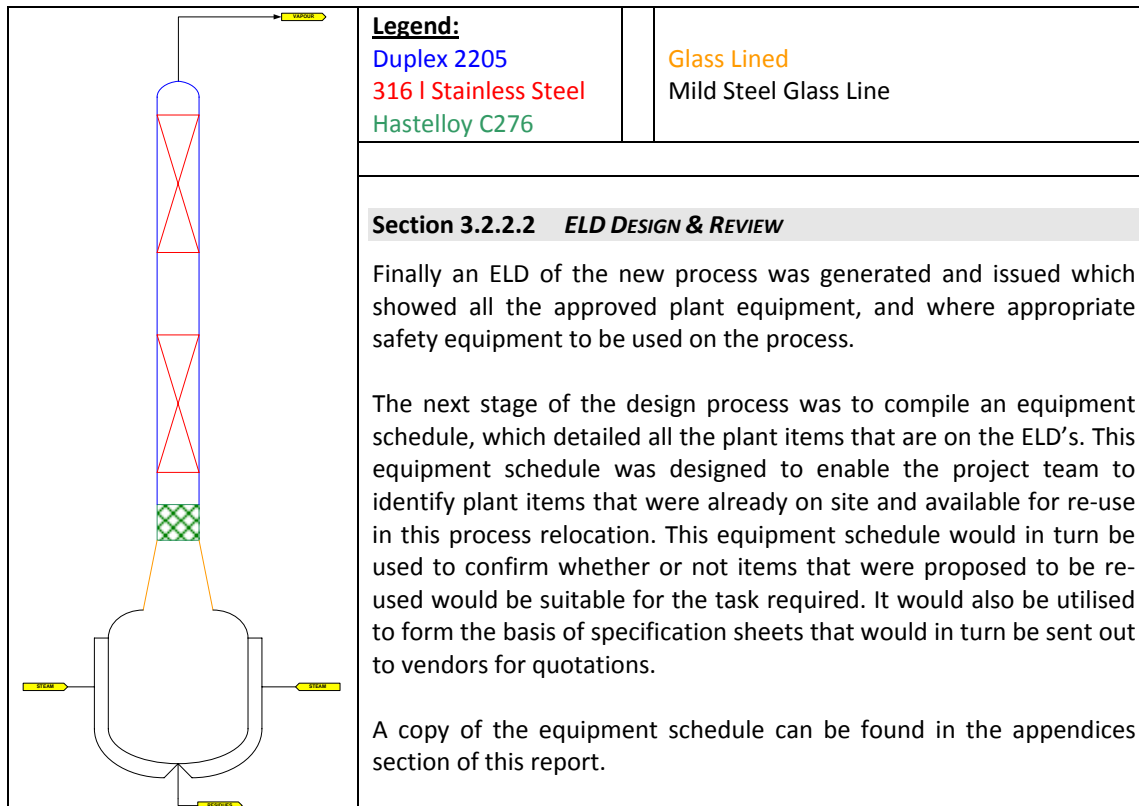


Figure 3.3: MOC diagram for new still pot and column.

Section 3.2.3 PHASE 3

Stages involved: -

1. Equipment schedule.
2. Process plant specification.
3. Detailed equipment design.
4. Services schedule.
5. Draft Control Philosophy.

Section 3.2.3.1 EQUIPMENT SCHEDULE

The next stage of the design process was to compile an equipment schedule, which detailed all the plant items that are on the ELD's. This equipment schedule was designed to enable the project team to identify plant items that were already on site and available for re-use in this process relocation. This equipment schedule would in turn be used to confirm whether or not items that were proposed to be re-used would be suitable for the task required. It would also be utilised to form the basis of specification sheets that would in turn be sent out to vendors for quotations.

A copy of the equipment schedule can be found in the appendices section of this report.

Section 3.2.3.2 PROCESS PLANT SPECIFICATION

Having completed this equipment schedule, it was possible to identify which piece of equipment would require either confirmation of capability, or design. These items were: -

- ✚ Still Pot (although this was only as far a liquid level within the pot, as the pot used was an existing strategic spare which was already on site).

- ✚ Column (for calculations referring to the column design please see appendices section).
- ✚ Main Condenser.
- ✚ Reflux Drum.
- ✚ Residue Cooler.
- ✚ Water Wash Column (this exercise was to ensure that the current column would be able to cope with the new duty placed upon it, as an existing wash column associated with a different process would be used for this process).

It was also necessary to confirm and size the following items: -

- ✚ Pumps to be re-used in the process.
- ✚ Pumps to be procured as new for the process.

When examining the existing pumps all that was required was a simple check of the pumps pump curve was required, so as to ensure the pump could deliver the head that was required. In the case of new pumps being specified, complete specification sheets needed to be generated. These specification sheets included, but were not limited to: -

- ✚ Material data.
 - Fluid properties.
 - Solids content.
 - Gas content.
 - Operating temperature.
- ✚ Flow capacity.
 - Designed flow rate.
 - Maximum required flow rate.
 - Range of flows.
- ✚ Suction conditions.
 - Pressure at equipment.
 - Design pressure at equipment.
 - Static head.
 - Suction line pressure drop.
- ✚ Discharge conditions.
 - Pressure at equipment.
 - Static head.
 - Pressure drop down stream of discharge point.
- ✚ Net Positive Suction Head (NPSH).

Copies of these specification sheets can be found in the appendices section of this report.

Section 3.2.3.3 *DETAILED EQUIPMENT DESIGN*

There were several items of equipment that require detailed design, the most notable of these items being: -

- ✚ Column (for calculations referring to the column design please see appendices section).

The out come of these calculations being that: -

- ✚ Column requires Theoretical Stages in order to perform the IPE/DMF separation.
- ✚ With a HETP varying from 0.25 to 1m, this results in a column height of 3.5 to 14m.
- ✚ For smallest height column, structured packing similar to that being used currently should be used.

Section 3.2.3.4 SERVICES ASSESSMENT

Using the data from the equipment schedule, detailed equipment design, and the known amounts of services currently used by the existing column, a site survey was completed so as to assess the service availability currently available in the area where the column was to be relocated. Details of the methodology used to carry out the assessment can be found in the appendices section of the report. The key results from the assessment can be found below: -

Service.	New Demand	Current Usage	Issue Status
Cooling Water.	1.2 te/hr	5.1 te/hr	None
Process Chilling Systems.	3.5 te/hr	3.9 te/hr	None
HP Steam.	1.2 te/hr	3.5 te/hr	None
Effluent Systems.	3.6 te/hr	5.5 te/hr	None

For calculations please refer to Appendix 8 – Service Assessment.

Section 3.2.3.5 DRAFT CONTROL PHILOSOPHY

A control philosophy is required in order to identify how the process is going to be controlled. Previously the recovery had been performed on a manual plant, i.e. no automation. With the new plant, automation was to be used in order to minimise process variability.

In order to compile a control philosophy a detailed knowledge of how the process operates is required. This detailed knowledge is then applied to the ELD of the process, and a logic path created (essentially a flow chart) which shows how the plant is to be operated. This flowchart details every operation involved in the process.

This draft control philosophy was generated by the area owner, who has over 10 years experience of running the process. This philosophy was then reviewed during phase 4.

Section 3.2.4 PHASE 4

Stages involved: -

1. HAZOP.
2. SIL Assessment.
3. Software Flowchart Review.

Section 3.2.4.1 HAZOP STUDY

A HAZOP study (HAZard and OPerability) study was conducted (during which I acted as scribe) with the aim of identify all the hazards associated with the process so that they could be designed out. In order to do this the process was subdivided into a series of nodes. Each node was then examined using a series of key words (or triggers). These key words being: -

- ✚ No.
- ✚ Reverse.
- ✚ More.
- ✚ Less.
- ✚ Part of.
- ✚ As Well As.
- ✚ Other.

These key words were applied to several parameters such as (Not all are applicable to all key words): -

- ✚ Flow.
- ✚ Level.
- ✚ Pressure.
- ✚ Temperature.
- ✚ Components.

- ✚ Vessel integrity failure

The main highlighted outcomes of the HAZOP review were: -

- ✚ Design control interlocks into the Control System.
- ✚ Ensure that the residue material is thermally stable below 260°C (this was done by lab analysis by one of our R&D sites).
- ✚ Identify the correct failure position for a number of air controlled valves.

Section 3.2.4.2 SIL ASSESSMENT

A SIL assessment was performed by the project team after completion of the HAZOP. Details on SIL assessment, and their methodology can be found in Appendix 10.

The outcomes of the SIL Assessment are detailed below, where the three scenarios numbered are: -

1. Overpressure leading to the rupture of the still, column or pipework.
2. Loss of reflux drum pump away leading to flooding of pipework and condensers, and the subsequent overpressure.
3. Overfilling of the still/column during batching.

Scenario	SIL Level	Outcome
1 – Personnel	1	This shows that specific reliability standards must be achieved based on reliability calculations carried out on the design.
1 – Asset / Production	2	
1 – Offsite	1	
2 – Personnel	-	This shows that there are no special safety requirements for the interlock system, i.e. the designer has complete freedom.
2 – Asset / Production	-	
2 – Offsite	-	
3 – Personnel	-	This shows that there are no special safety requirements for the interlock system, i.e. the designer has complete freedom.
3 – Asset / Production	-	
3 – Offsite	-	

Section 3.2.4.3 SOFTWARE FLOWCHART REVIEW

This stage of the design process focusses on review the control philosophy flowchart described earlier in this report. The review centred around ensuring that all the flowcharts detailed what they were intended to do, and that they performed these tasks in the most efficient way.

Section 3.2.5 PHASE 5

Stages involved: -

1. Procurement.
2. Construction.
3. Commissioning.
4. Handover.

Section 3.2.5.1 PROCUREMENT

During this phase the key activity was liaising with suppliers so as to ensure that the quotations they provided were accurate which would enable the project team to make an accurate and fair assessment of which supplier to place orders with.

Section 3.2.5.2 CONSTRUCTION, COMMISSIONING & HANDOVER

The final parts of this project relate to the actual construction, commissioning and final handover to the area. At the time of writing this report these activities were not complete as I was due to be transferred to another site prior to their initiation.

Little assistance is required during the construction phase as this is subcontracted out to other parties, and is controlled via the project leader.

However some involvement came in the generation of commission programmes for the various parts of the project. The stages of commissioning are detailed below: -

- ✚ ELD Checks
Ensure that the plant has been constructed as the ELD states.
- ✚ Line labelling.
Label all lines to ensure ease of understanding for operators etc.
- ✚ Flushing.
Flush all lines and vessels to remove stray debris.
- ✚ Vessel Calibration.
Ensure that all controllers function properly (i.e. level, flow, pressure etc)
- ✚ Control systems checks.
Ensure that what the control system claims are being performed, it actually being done on plant.
- ✚ Tank checks.
- ✚ Water Simulations.
Run the process using only water, this is done so as to ensure all levels etc are accurate etc.
- ✚ Solvent Simulations.
Perform the process using clean solvents; this is done so as to ensure that the number of theoretical stages in the packing is as the manufacturer states it should be.
- ✚ Process Commissioning
Column is run using actual process fluid, and assistance is provided for any trouble encountered.

At handover, a full detailed commissioning report is presented to the area owner detailing all the tests performed on the process, the results of these tests, and remedial actions taken to alleviate any problems encountered.

Section 3.3 ESTIMATE OF TIME DEVOTED TO THE WORK

Involvement with this project ran from September 2007 to September 2008, with work for the bulk of that period being taken as a full working day, 3.5 days a week, resulting in a total time dedicated of: -

182 days (1456 hrs) [approximately 6 months]

¹ No associated cost with water as site has its own bore hole, which is used to supply water.

² No associated cost for air, as this is a result of leaks into the column when under vacuum.

³ No associated cost for salts and solids as these are impurities found within the main components.

⁴ Values correct as of 13-August-2008, sourced from [REDACTED] Procurement

⁵ A simplified diagram of the original process can be found in the appendices section of this report.

Section 4. CONCLUDING STATEMENT

During the undertaking of this project, there have been many areas in which I feel my skill set have been greatly enhanced. These areas include: -

Understanding of distillation column design.

In undertaking the design of this column it has been necessary to investigate further the concepts of distillation, the modes of operation (i.e. continuous or batch), and to investigate the area of packing in columns (both random packing such as Pal Rings, and structured packing).

HAZOP study.

Previously I had never attended a HAZOP session before, during the sessions associated with this project I not only attended and contributed, but also scribed, thus gaining a great insight into the HAZOP process. I also attended training sessions relating to HAZOP prior to taking part in the assessment.

SIL Assessment.

As with HAZOP I had no previous experience of SIL Assessments, but now am familiar with the process behind the SIL Assessment, and the purpose of the assessment and the intended outcomes.

Surveying work.

Prior to commencing this project I had no experience of surveying work, including line sizing (both diameter and run length including bends etc), and also surveying these lines in terms of suitability for the duty they were proposed to be assigned.

Working as part of a large multi-disciplinary project team.

Previous projects within university environment had all been single discipline teams of all chemical engineers. Being part of a large multi-disciplinary team enabled me to gain a better understanding of what different professions contribute to projects, and the constraints they find themselves under during the project process. To this end I now feel I am better equipped to be able handle these groups when issues arise, and keep the group focused on the critical path and enable the project to move towards completion.

Process simulation.

Previous process simulation experience had been done using Chemstations ChemCAD ©, and AspenTech Hysys ©, during the undertaking of this project I have had to learn how to use new software packages including AspenTech Aspenplus ©, AspenTech BatchSEP © and ABB PEL Suite ©. I have now demonstrated competence in using these software packages to complete numerous modelling simulations during this project.

Economic appreciation of distillation's value to industry.

As the economic constraints on the project have been quite significant due to external pressures on both the company and the site, I have gained a good level of experience with regards to taking into account the financial implications of decisions made on the project. To this end the project made several key decisions in order to manage the cost. These decisions included (but were not limited to): -

- Introducing a disentrainment section so as to prevent liquid carryover into the column and packing, allowing the column and packing to be made out of non-exotic materials.
- Where possible the project team looked to re-use equipment that was already onsite but was now redundant due to other projects that were ongoing on site.

FURTHER LEARNING TO MASTERS LEVEL REPORT	VERSION 1.0
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Section 5. ATTESTATION

This is to attest that the contents of this report are an accurate representation of the work carried out in the completion of this project.