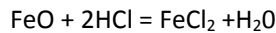


Appendix C3 Appendix 2 1 – technical descriptions

Process 5

Appendix C3 Appendix 2 1 a details the materials balance for both processes 5 & 6. The production of ferrous chloride, is a simple one. Ferrous chloride is produced by dissolving iron-scale (mill-scale)(FeO) in 25% Hydrochloric acid (at this strength there are no fumes & no requirement for a scrubber unit). The following reaction occurs:-

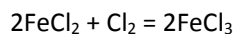


The Iron source is added to the dissolvers using a hoist & is always kept in excess by dipping to check the bed level.

25%HCl is received by the plant & held in a feed tank. The HCl is then pumped from the feed tank to the dissolvers. The liquid is then circulated to enable the HCl to dissolve the Iron. Once reacted the ferrous chloride solution overflows into the recycle tanks to be filtered and a passed through a cooler to control the temperature. At this point the ferrous chloride is either distributed to the customer or sent to the Ferric chloride plant (process 6) to be oxidised further to convert the Fe²⁺ to Fe³⁺ and create ferric chloride.

Process 6

The manufacturing process for the production of ferrous chloride was previously detailed in permit EP3635TU (currently operated at the Runcorn facility). The manufacture of ferric chloride will be a continuation of the ferrous process. Once the ferrous chloride has been manufactured, the product is tested to determine the strength of the product. If it is to be sold as ferrous chloride then it will be passed through a filter-press to remove any undissolved solids and is then sent to storage. However if it is to be used to manufacture ferric chloride the ferrous chloride is feed into a ferric tower, will is packed with mill-scale (a form of iron oxide) and as it passes through the tower, chlorine gas is fed in from the opposite end. This is to enable the chlorine to chlorinate the ferrous chloride to ferric chloride and also to increase the iron content of the product. The once the product has been through the ferric column, it is sampled and tested to determine its strength, if it is at the required strength, it is then passed through a heat exchanger to bring the temperature down, then it is pushed through the filter press and then sent to storage. However if the strength is not at the correct level, it is then is passed through heat exchangers to bring the temperature of the product down put back through the ferric column. Both the manufacture of ferrous and ferric chloride are exothermic reactions and as a result of this, the product requires cooling to prevent the removal of the hydrochloric acid or the potential for ferrous/ferric hydroxide to be precipitated by hydrolysis.



Process 7

The sulphuric acid is passed through the evaporation system as seen in Appendix C3 Appendix 2 1b. The increased strength Sulphuric acid is sent to storage. The condensate is then treated and sent off-site for disposal. There is no change to the chemical formula and Appendix C3 Appendix 2 1b illustrates the process.

Process 8

Aluminium chloride (AlCl_3), aluminium chlorohydrate ($\text{Al}(\text{OH})_a\text{Cl}_b$ where $a + b = 3$) and polyaluminium chloride ($\text{Al}(\text{OH})_a\text{Cl}_b(\text{SO}_4)_c$ where $a+b+2c = 3$), commonly known as PAC.

These compounds are prepared by reacting aluminium hydroxide ($\text{Al}(\text{OH})_3$), also known as alumina trihydrate, with hydrochloric acid in heated reactors with, in the case of PAC, the addition of sulphate either as sulphuric acid or as a sulphate salt.

PAC is not a stoichiometric compound and requires an excess of aluminium hydroxide to drive the reaction to completion. This excess is filtered off and because, at this time, it cannot be recycled, it is sent off site to be disposed. After filtration the solutions are "trimmed" to the desired strength by adding water.

The only volatile substance involved in these processes is hydrochloric acid and the emission of vapours from this is controlled by the use of a single pass scrubber where the effluent passes to a storage tank and is used to dilute the basic products to the required strength.

Appendix C3 Appendix 2 1 c shows this process taking place.

Process 9

The production of Sodium silicate is produced by the classical hydrothermal procedure of heating "glass" grade silica sand (quartz) and sodium hydroxide solution in pressure reactors. Because few silicates are stoichiometric compounds each requires an excess of sand to drive the reaction to completion. This excess is filtered off and re-used until such time that it is no-longer suitable to be used as a starting material in the reaction. At this point it will be removed from the system and treated onsite to form a neutral solid suitable for non-hazardous disposal. This process is currently operated under permit DP3637SG and due to process refinements produces significantly less waste than was originally projected in DP3637SGG original permit application.

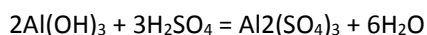
Appendix C3 Appendix 2 1 d shows this process taking place

Process 10

This is also a very simple process and is detailed in Appendix C3 Appendix 2 1 e

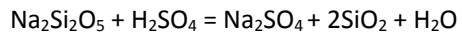
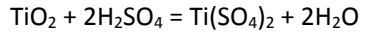
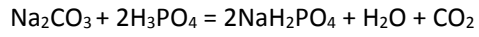
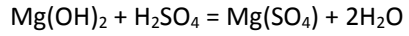
Process 11

This solution is made by reacting aluminium hydroxide with diluted sulphuric acid in an acid resistant reactor, filtering off any unreacted residue (and used in the next batch as a starter), trimming the solution to the desired strength by adding water. At this time the only reaction carried out is between the aluminium hydrate and the sulphuric acid to produce the aluminium sulphate solution:-

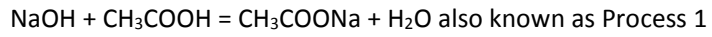


This process uses the heat of dilution of the sulphuric acid to initiate the reaction and the energy from the exothermic reaction of an acid with a base makes the process self-sustaining. Appendix C3 Appendix 2 1f illustrates this.

However it is envisaged that this will be the primary plant that with little or no modifications can be used to act using the Multi Product Protocol. The product range would be primarily non-volatile raw materials, using non-toxic acid reacted with a non-toxic base. The examples detailed below which include but are not limited to:-



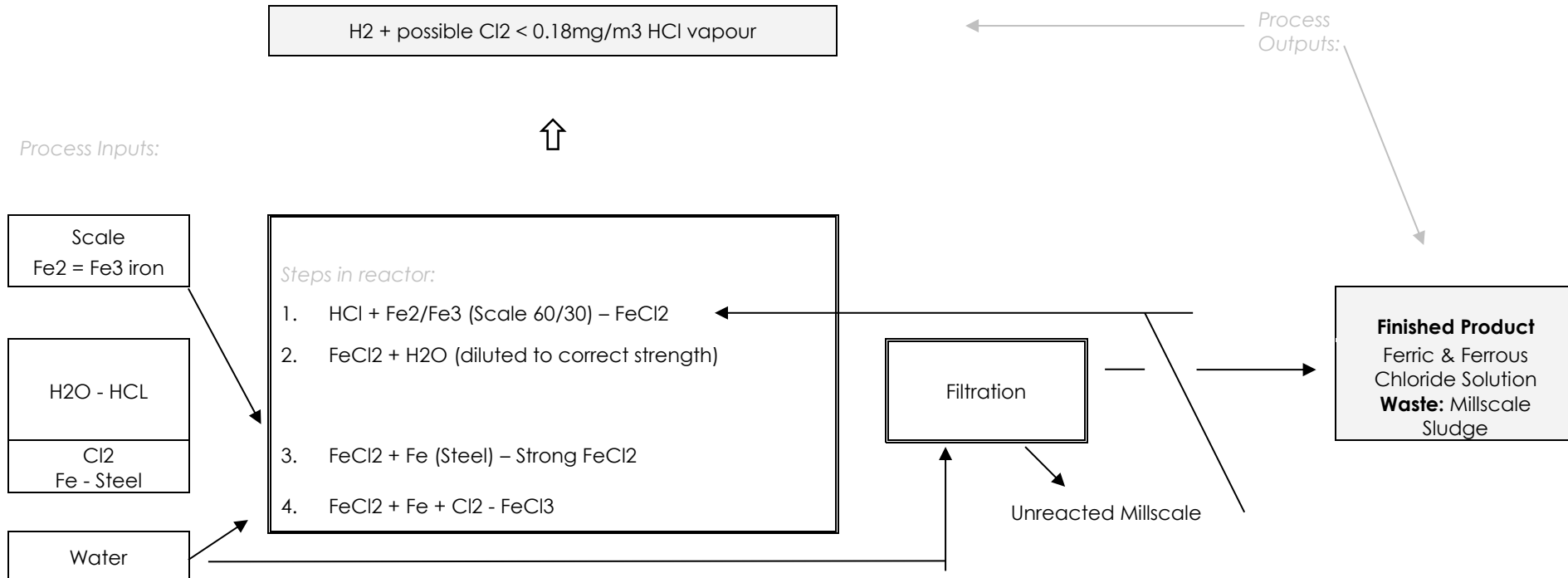
And with minor modifications:-



Material Balance for Ferrous & Ferric chloride manufacture – Runcorn Iron Salts

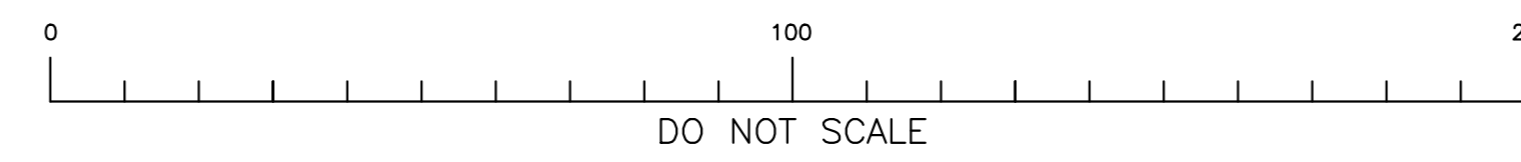
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Ferrous & Ferric chloride Production:

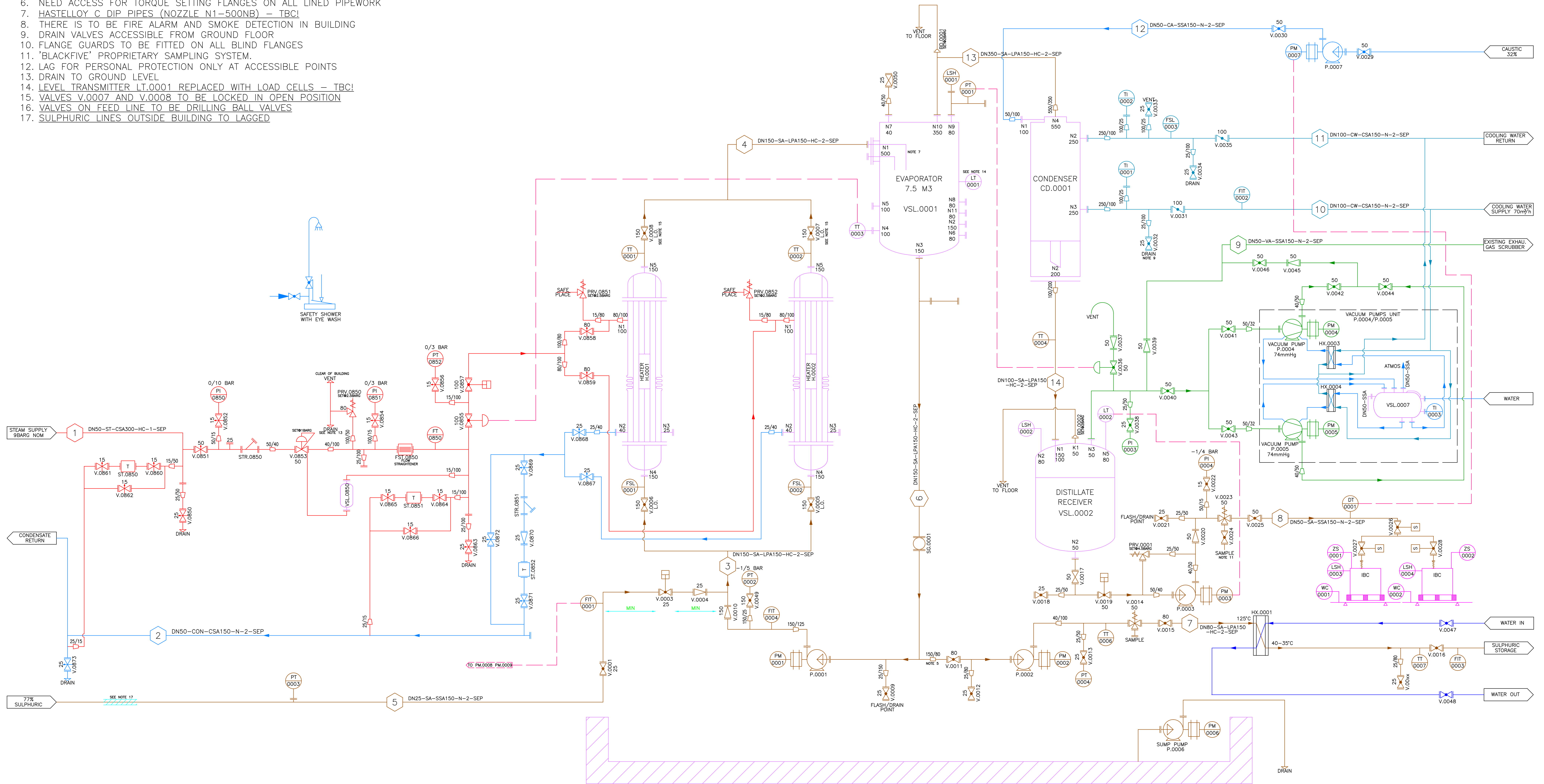


- Notes
- Millscale a mixture of F₂+F₃ iron added to 24% HCl to make 70% FeCl₂ / 30% FeCl₃.
 - H₂O added to FeCl₂ reducing the density for greater control of the ferric chloride process.
 - FeCl₂ + Fe (Steel) circulated through a dissolver to increase the FeCl₂ strength for accurate FeCl₃ conversion to 38.5 / 42.0% FeCl₃.
 - Ferric Chloride produced from addition of Cl₂ to strong FeCl₂ = FeCl₃ 38.5 / 42.0%.

Changes to this issue: First Issue					
Originator	Title	Date	Approvals	Title	Date
 Paul Price	Site Manager	4 th January 2017	 Claire Harling	Environmental Manager	4 th January 2017
Issue: 1	Effective Date: 04/01/2017		ICL-EMS-RUN-DOC5030	Form 1009	Page 1 of 1



- ALL BENDS AND TEES TO BE REMOVABLE, ADDITIONAL FLANGES TO BE INSTALLED IF NECESSARY
- PIPEWORK TO BE DESIGNED TO REDUCED ENTRAINMENT / CARRY OVER
- THE LEVEL IN THE EVAPORATOR IS CONTROLLED BY VARYING THE PRODUCT TRANSFER RATE TO THE STORAGE TANKS
- SPEC CHANGE - DIP PIPE IN HASTELLOY C - TBC!
- ECCENTRIC REDUCER B.F.
- NEED ACCESS FOR TORQUE SETTING FLANGES ON ALL LINED PIPEWORK
- HASTELLOY C DIP PIPES (NOZZLE N1-500NB) - TBC!
- THERE IS TO BE FIRE ALARM AND SMOKE DETECTION IN BUILDING
- DRAIN VALVES ACCESSIBLE FROM GROUND FLOOR
- FLANGE GUARDS TO BE FITTED ON ALL BLIND FLANGES
- 'BLACKFIVE' PROPRIETARY SAMPLING SYSTEM.
- LAG FOR PERSONAL PROTECTION ONLY AT ACCESSIBLE POINTS
- DRAIN TO GROUND LEVEL
- LEVEL TRANSMITTER LT.0001 REPLACED WITH LOAD CELLS - TBC!
- VALVES V.0007 AND V.0008 TO BE LOCKED IN OPEN POSITION
- VALVES ON FEED LINE TO BE DRILLING BALL VALVES
- SULPHURIC LINES OUTSIDE BUILDING TO LAGGED



HAZOP COMMENTS INCLUDED										P.J. 04/10/11										P.J. 12/07/11										P.J. 12/07/11														
FIRST ISSUE										REVISIONS										REVISIONS										REVISIONS														
ISSUE	EC NUMBER	REVISIONS	DRAWN	DATE	CHECK	DATE	PROJ	DATE	MECH	DATE	ELECT	DATE	CIVIL	DATE	ISSUE	EC NUMBER	REVISIONS	DRAWN	DATE	CHECK	DATE	PROJ	DATE	MECH	DATE	ELECT	DATE	CIVIL	DATE	ISSUE	EC NUMBER	REVISIONS	DRAWN	DATE	CHECK	DATE	PROJ	DATE	MECH	DATE	ELECT	DATE	CIVIL	DATE

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SCALE: NTS

TITLE: SULPHURIC ACID CONCENTRATOR

INDUSTRIAL CHEMICALS GROUP LTD

TPSSE 05794

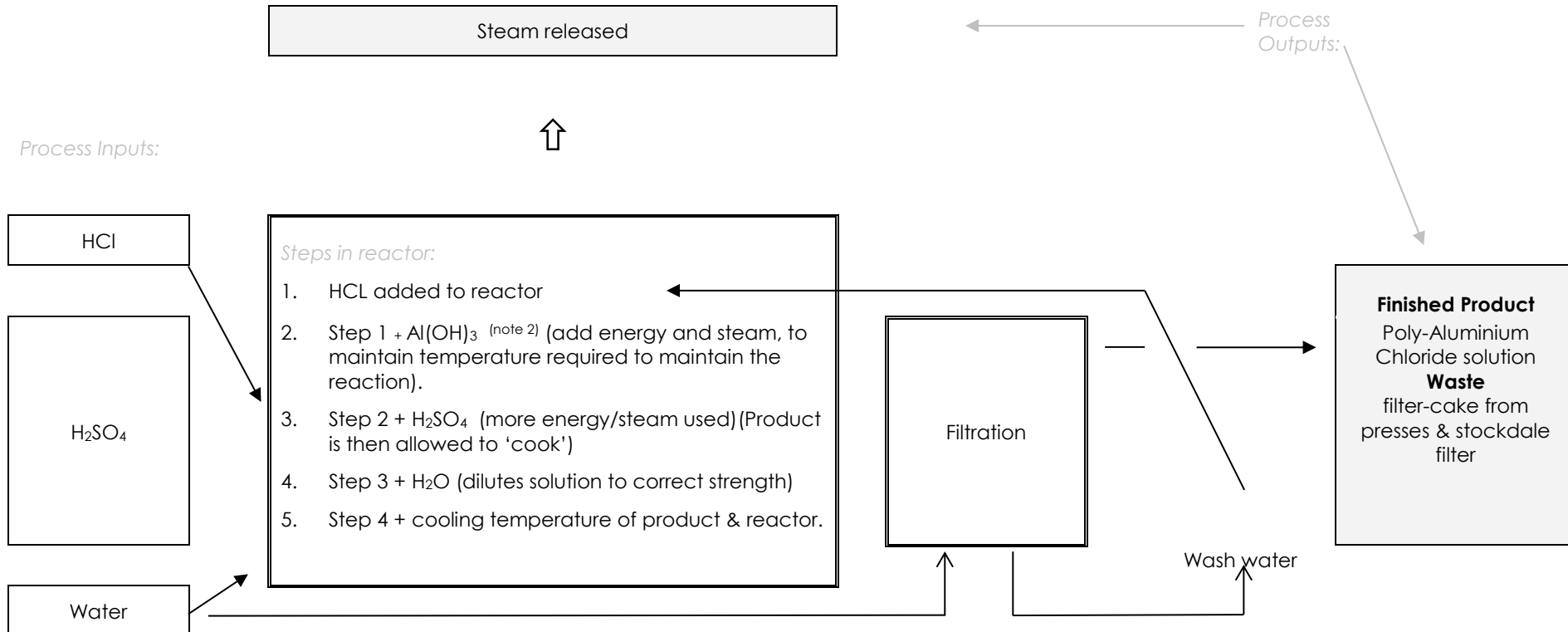
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ISSUE: B

Material Balance for Poly-Aluminium chloride manufacture – Grays

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Poly-Aluminium chloride Production:



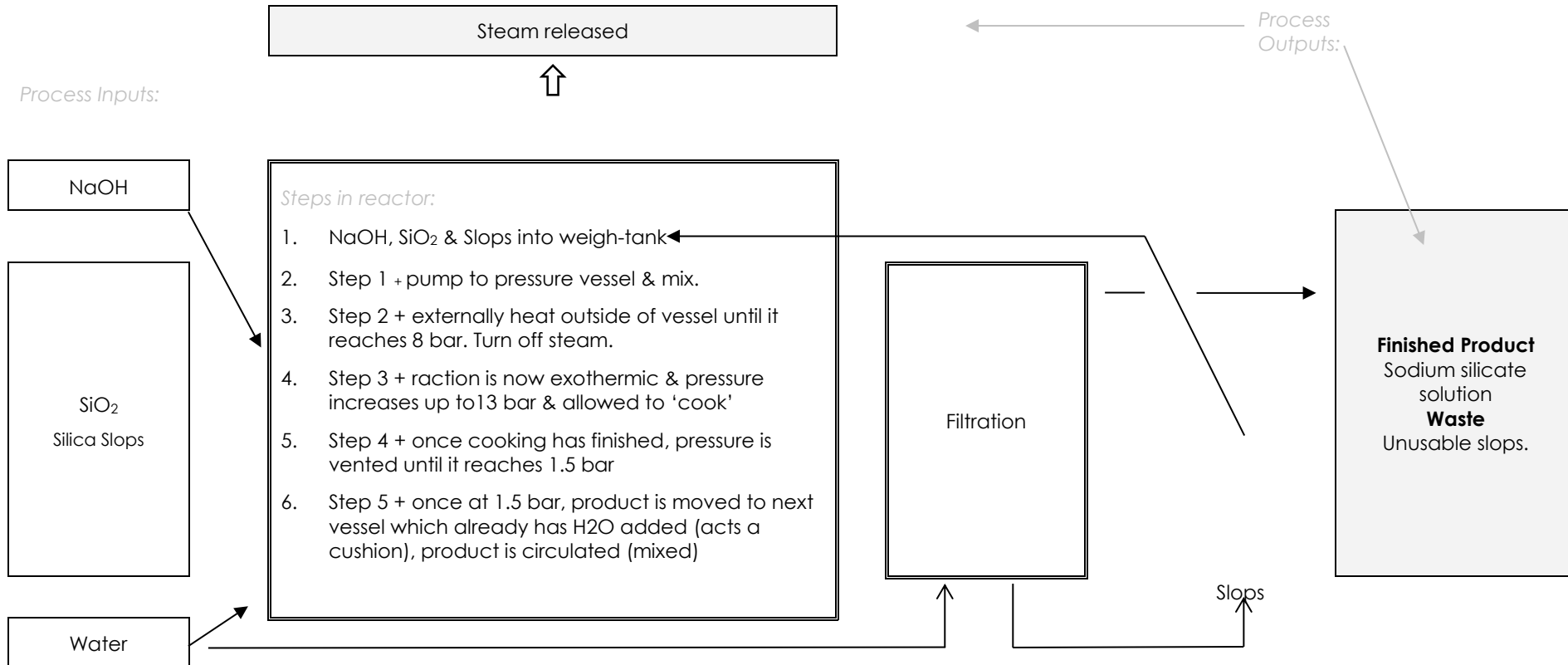
- Notes
- The filtrate is back washed into the reactor creating the initial charge for the next batch (step 4) to create dilution water.
 - Al(OH)₃ is added in excess at 0.1-0.2% as part of the process control strategy.

Changes to this issue: Document Review – No changes required					
Originator	Title	Date	Approvals	Title	Date
 Claire Harling	Environmental Manager	2 nd November 2016	 Tony Peak	Plant Manager	2 nd November 2016
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Material Balance for Sodium silicate manufacture – Grays

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Sodium silicate Production:



Changes to this issue: Document Review – No changes required					
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Tri-sodium Citrate Plant Operating Procedure

TSC-OP001: Tri-sodium Citrate Production and Out-loading

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1.00 PURPOSE AND SCOPE

This document details plant operating procedures for producing and out-loading Tri-sodium citrate solution.

2.00 DEFINITIONS

None

3.00 REFERENCES

None

3.01 Supporting Documents

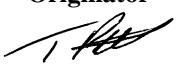

Form 0007: Goods Inwards Acceptance Form – Tri-sodium Citrate solution
 Form 0034: In-Process Testing Schedule – Tri-sodium Citrate solution
 Form 0070: Final Q.C. Sampling and Analysis Schedule – Tri-sodium Citrate solution
 Form 0072: Starting Materials Specification List – Tri-sodium Citrate Solution
 Form 0244: Tri-sodium Citrate Batch Sheet
 Form 0296: Formula Card – Tri-sodium Citrate Solution

4.00 PROCEDURE

Process Overview and Quality Concerns

4.01 Tri-sodium citrate solution is produced from softened water, Citric acid solid, and Caustic soda liquor (which is also called Sodium hydroxide solution) in a dedicated vessel.

Due to high water and organic matter content, and mid-range pH, the finished solution is susceptible to microbiological contamination. The production process therefore uses the acidity of Citric acid and the alkalinity of Caustic soda to create low and then high pH, with the intention of destroying any bugs that may be present in the water and / or Citric acid used. The batching vessel is also cleaned with hot water before use as an additional microbiological control.

Changes to this issue: Document Review					
Originator	Title	Date	Approvals	Title	Date
 Tony Peak	Plant Manager	25 th Aug 16	 Peter Jordan	QA Manager	25 th Aug 16
Issue: 2	Effective Date: 25/08/2016		ICL-QA-TSC-DOC2058		Page 1 of 5

Tri-sodium Citrate Plant Operating Procedure

TSC-OP001: Tri-sodium Citrate Production and Out-loading

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4.02 The production sequence is as follows:

Softened water is added to the batching vessel; most of the Citric acid required is dissolved in the water; sufficient Caustic soda is added to make the batch strongly alkaline; the remainder of the Citric acid is dissolved; the batch is trimmed with Caustic soda or Citric acid.

4.03 Tri-sodium citrate shall be made to order and out-loaded for customer delivery from the batching vessel.

4.04 Note that the final product has an alkaline pH due to a small excess of Caustic soda. Experience has shown that the pH can change after production as the small excess of Caustic soda reacts with carbon dioxide from the atmosphere. pH must be checked just before out-loading, and may need to be adjusted.

Safety Notes

4.05 Caustic soda is corrosive and will cause severe skin burns and severe eye damage. Wear standard site personal protective equipment. Wear appropriate gloves when taking samples.

4.06 The reaction between Citric acid and Caustic soda is strongly exothermic.

Batch Sheets, Formulation, and Sampling & Analysis

4.07 Keep production records using Form 0244: Tri-sodium Citrate Batch Sheet.

4.08 Take samples as directed on:

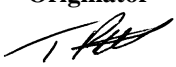

Form 0007: Goods Inwards Acceptance Form – Tri-sodium Citrate solution

Form 0034: In-Process Testing Schedule – Tri-sodium Citrate solution

Form 0070: Final Q.C. Sampling and Analysis Schedule – Tri-sodium Citrate solution

4.09 Use the grades of starting materials specified on Form 0072: Starting Materials Specification List – Tri-sodium Citrate Solution.

Quantities to be used are specified on Form 0296: Formula Card – Tri-sodium Citrate Solution.

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Tri-sodium Citrate Plant Operating Procedure

TSC-OP001: Tri-sodium Citrate Production and Out-loading

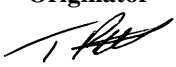

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Hot Water Cleaning

- 4.10 Before the batch process can commence it is essential that the vessel is hygienically cleaned:
1. Fill trailer from vessel 515 (containing softened or evaporated water), with 20 tons of water to commence hot water cleaning process.
 2. Heat water to above 90°C. Note that this can take between 16 to 18 hours.
 3. Transfer the hot water from the road barrel to the citrate vessel and allow to circulate throughout the system for a minimum of 90 minutes. Temperature should be monitored at least every 30 minutes to ensure it does not fall below 80°C.
 4. Record final temperature before transfer of water from citrate vessel back to effluent via loading line. (The loading line will also be hot water cleaned during this process.)
 5. Record the time at which the citrate vessel was emptied of the hot water.
 6. Within 2 hours of the citrate vessel being emptied of the hot water it is important that the citrate batching process begins, otherwise the citrate tank will require a second hot water hygienic clean.

Batching Process

- 4.11 Note that the batch size is set so that one batch equals one load, with weight of additions determined by the formula card.
- 4.12 Assuming the condition set in 4.10 point 6 is met, add the volume of softened water specified from the formula card to the citrate batching vessel. Record the time of commencing the batching process.
- 4.13 Begin mixing / external circulation using pump including agitation in vessel.
- 4.14 Observe the temperature from the citrate vessel panel and ensure that it is greater than 15°C. If temperature is too low the vessel will need to be emptied and refilled (below this temperature the Citric acid will not dissolve).
- 4.15 The pH of water is required to be between 7 and 10.

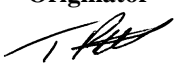

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Tri-sodium Citrate Plant Operating Procedure

TSC-OP001: Tri-sodium Citrate Production and Out-loading

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- 4.16 Add the first required citric addition indicated from the formula card to the citrate vessel, using the “Spiroflo” system and the following ‘lifting plan’:
- Lift a bag above the feed hopper for the elevating screw, making sure that each bag handle is secure on the lifting frame ‘spider’ and that the path of the bag when elevated is free from obstructions / personnel, etc. Before lowering untie the bag outlet and start the screw. To ensure that the bag empties, it may be necessary to slightly lift the bag as it empties. Repeat until the initial charge of Citric acid has been added. Ensure that the screw is turned off while untying each bag bottom. Note that it is counterproductive to add the Citric acid too quickly as a large quantity of undissolved solid may cause the agitator to trip.
- 4.17 Allow the Citric acid solution mix to circulate for 30 minutes to ensure total dissolution.
- 4.18 Submit sample for analysis for pH. If greater than 2.5, allow circulation for a further 30 minutes and resample, if pH is still greater than 2.5 the laboratory will advise any further adjustments.
- 4.19 Add the required volume of (36%) Caustic soda liquor using the caustic pump.
- 4.20 Record temperature after 30 minutes.
- 4.21 Submit a sample to the laboratory for pH analysis. If pH is less than 12.9, add additional caustic as instructed by the laboratory.
- 4.22 Add the remainder of the Citric acid as specified from formula card.
- 4.23 Recirculate for 2 hours.
- 4.24 Submit a sample to the laboratory (for density [s.g.], appearance and pH determination).
- 4.25 Follow any advice to trim the batch to specification but also note that any further addition of water may only be added with the contents of the citrate vessel at a pH greater than 11 and at a temperature in excess of 80°C. (Two stages may be necessary – water addition to achieve the correct density / s.g., and then Caustic soda or Citric acid addition to achieve the correct pH.)

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Tri-sodium Citrate Plant Operating Procedure

TSC-OP001: Tri-sodium Citrate Production and Out-loading

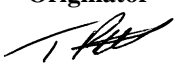

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- 4.26 Assuming an addition is necessary, recirculate for 1 hour (minimum) before resampling to confirm that the adjustment has achieved conforming product. (Return to step 4.24 as necessary)

Out loading to Tanker

- 4.27 Once product is ready to load, submit a final sample for analysis. If no adjustment is required a certificate of analysis will be issued and the lot is available for release with a dedicated lot number and QC tag. The designated lot may then be loaded into a sanitised tanker. If an adjustment needs to be made prior to out loading a final sample shall be submitted for analysis to obtain a certificate of analysis.
- 4.28 Trailer sanitisation certification must not be older than 8 hours. Certification for the tanker must be checked by the loading operator prior to out loading of the Tri-sodium citrate.
- 4.29 Take seven final hygienic samples (aseptic samples), including one for pH measurement, of the finished and loaded Tri-sodium citrate for submission to P&G after loading. Samples should be labelled according to sampling procedure TSC-OP002.
- 4.30 Seal Trailer outlets.

NB: To maintain the pH > 11 Citrate should be transferred to the tank-car immediately after making from the Citrate Vessel and then stored in the sealed tank-car (pending positive Micro release of sample) for a maximum of 14 Days.

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