

PRIFYSGOL CYMRU

Y Drindod Dewi Sant

UNIVERSITY OF WALES

Trinity Saint David

School of Engineering

Dissertation

Independent Project

ACMA 6013

*An Investigation to verify Sulphur content in Nickel Sulphate Hexahydrate for
the Hydrogen Reduction of Nickel Oxide*

Paul Benbow

2014803

17th May 2024

This thesis is the result of the author's original research. It has been composed by the author and has not been previously submitted for examination which has led to the award of a degree.

Signed:  Date: 17/5/24.

Abstract

This report attempts to identify opportunities that may improve the efficiency of a continuous chemical process which uses equipment that consumes Natural Gas. The primary reason for this topic was to establish financial savings which may defend profit margins against the rise of fixed costs associated with manufacturing, helping to ensure the longevity of the division. However, the decline of the Steel Industry in South Wales soon provoked a realisation that a business can no longer rely on profitability for longevity but, must also consider the impact it has on the Environment, this reassured the author that the topic selected was appropriate.

The 'Research and Literature survey' of this report focuses on a subject matter that is relevant to the topic and, was used to learn the function of each process step as well as their chemical kinetics. In doing so, provided the knowledge for when using a suite of continuous improvement tools to identify that; a deficiency in Sulphur may be the cause of this reports problem statement.

Analytical method were used to estimate the current proportion of Sulphur added to a Hydrogen Reduction process then, samples taken to perform laboratory experiments to find the actual quantity of Sulphur. Our results were inconclusive because we deemed our sampling plan flawed however, we observed a consistently higher percentage of Sulphur to that of the value calculated using the analytical method.

Our further work shall involve the implementation of a process sampling plan in contrast to the population sample plan that this report has employed and, we shall also pursue further investigations which shall be based on our results that; have indicated that there may exist a relationship between the pH level of an additive used to deliver Sulphur into the process and, the effectiveness of the Hydrogen reduction process.

Table of Contents

Abstract.....	ii
Table of Contents.....	iii
List of Figures	iv
List of Tables	iv
Glossary.....	iv
1. Introduction	1
1.1. Aims & Objectives.....	2
2. Timing Plan	3
3. Research & Literature Survey.....	5
3.1. The Process at Clydach	5
3.2. The Reduction & Activation Step.....	5
3.3. Theory of the Reduction Process.....	9
3.4. Theory of the Activation Process.....	10
3.5. Volatilisation Step.....	10
3.6. Theory of the Volatilisation Step	11
3.7. Problem Statement	11
3.8. Previous Investigations.....	14
4. Investigation.....	15
5. Experimental Findings	22
6. Discussion and Conclusion	23
7. Further Work.....	27
Bibliography	30
Appendix A	33
Appendix B	50
Appendix C	53
Appendix D	65
Appendix E	71
Appendix F	75
Appendix G	86

List of Figures

Figure 1, Gantt Chart of Project Milestones	4
Figure 2, Inputs and outputs of Reduction and Activation Step (Vale Europe Ltd., 2023)	7
Figure 3, Process Flow up to and, including the Reduction & Activation Step	8
Figure 4, Inputs and outputs of the Volatilisation Step (Vale Europe Ltd., 2024)	11
Figure 5, Trend of Natural Gas consumption relative to quantity of TNOS processed through Kiln Line 1 (Sterling, 2023).	12
Figure 6, Trend of Natural Gas consumption relative to quantity of TNOS processed through Kiln Line 2 (Sterling, 2023).	13
Figure 7, Comparison between Kiln 1 and Kiln 2 Born Heater's Exit Hydrogen Temperature Setpoint (Foxboro DSC, 2024).....	13
Figure 8, Comparison between Kiln 2 Hydrogen temperature setpoint and, Kiln 2 %Unreduced value (Foxboro DSC, 2024).....	13
Figure 9, Impact Matrix evaluating causes of the problem statement (Croft, 2024)	15
Figure 10, Cause and Effect diagram used to establish potential reasons why Kiln 2 Born heater consumes additional Natural Gas	16
Figure 11, The Relationship between Nickel Sulphate Type and % Unreduced Nickel.....	17
Figure 12, 5-Whys exercise to identify root causes.....	17
Figure 13, Photograph of rate parameters for a 25:1 feed ratio for a total feed rate between 2,000 and 3,300 kg per hour.....	19
Figure 14, Photograph of rate parameters for a 25:1 feed ratio for a total feed rate between 3,400 and 4,600 kg per hour.....	20
Figure 15, Control Chart for Sulphur Content in Nickel Sulphate Hexahydrate	27
Figure 16, Identification of proposed and current sample point in the Reduction and Activation Step	28
Figure 17, Photograph of the X-ray Fluorescence Spectrometry analyser used during the experiment.....	50
Figure 18, Nickel Sulphate Hexahydrate samples taken from four individual consignments	50
Figure 19, Photograph of samples dissolving on a hotplate.....	51
Figure 20, Photograph of sample receptacles which are loaded into the X-ray Fluorescence Spectrometry analyser	51
Figure 21, Photograph of receptacles before loaded into the X-ray Fluorescence Spectrometry analyser	52
Figure 22, Photograph of Metler Toledo pH controller.....	52
Figure 23, A photograph of the apparatus described in our further work to establish the effects of pH on the Hydrogen Reduction process	86

List of Tables

Table 1, Total mass weight of each element contained in Nickel Sulphate Hexahydrate (Chemical Aid, 2024) ..	19
Table 2, Test Results (Jayne, 2024)	22
Table 3, Average Percentage of Nickel that was unreduced during a campaign of Nickel Sulphate Hexahydrate manufactured by Umicore	25
Table 4, Average Percentage of Nickel that was unreduced during a campaign of Nickel Sulphate Hexahydrate manufactured by Bluestone.....	25

Glossary

Term	Definition
TNOS	Tokyo Nickel Oxide Sinter, the primary material used in the process
$\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$	Nickel Sulphate Hexahydrate, an additive used in the Hydrogen reduction process
SO_2	Sulphur Dioxide, a gas used in the activation Kiln
H_2S	Hydrogen Sulphide, a gas produced in the Activation Kiln

1. Introduction

The conflict between Russia and Ukraine exposed the UK's vulnerability for relying on an imported Natural Gas supply, as sanctions were imposed on Russia global demand for the commodity rose. During 2022 and 2023 the wholesale cost of Natural Gas in the United Kingdom increased by 404% (Jackman, 2023), inflating the fixed cost's associated with manufacturing processes, placing the UK's manufacturing sector at a disadvantage to their foreign competitors. Vale's operations in Clydach were subject to this very scenario, with processes that used large volumes of Natural Gas for steam reforming into Hydrogen and, as fuel to heat furnaces that were required through-out their manufacturing process.

The increase to fixed costs led to intensified internal competition with Vale's Copper Cliff Nickel refinery in Ontario, Canada. The competition was based on who could produce a Nickel product which had the largest profit margin, the price would be; which refinery received the Nickel Oxide mined in Indonesia. Historically, the refinery in Clydach would produce a premium product which could be used for specialist application such as Nickel alloys, Stainless Steel and Electric Vehicle batteries (Dalling, 2019). Nevertheless, during this period the refinery in Ontario had significantly lower fixed costs, with a comparison showing that Canadian business consumers pay 72% less for their Natural Gas than their UK peers (GlobalPetrolPrices.com, 2023), with the extra operational costs imposed on the Clydach refinery being absorbed by the premium product's profit margin. This scenario awoke the realisation that if a healthy profit margin could not be maintained the benefit of the organisations refining Nickel in the UK would be lost, although the cost of Natural Gas has since stabilised in the UK, the decision of the organisation remain steadfast in finding reduction to their Natural Gas usage.

The initial stages of this project coincided with the announcement from TATA Steel that they were to close their steel production facility in Port Talbot, it was reported that the Indian owned company no longer believed that producing steel using an environmentally unfriendly method was sustainable (Pigott & Price, 2024), this prompted the leadership team at Clydach's refinery to reflect on their Environmental, Social and Governance (ESG) policy, leading to a further endorsement of initiatives which could demonstrate a continuous improvement regime that was not limited by increasing profitability by lowering resource waste but, also considers the impact that it's business has on the environment. Fulfilling Vale's ethical obligations to the environment would provide economic benefit in the form investment from stakeholders who require assurance that their funds would be secure, evidence of this would be provided with certification from an international quality standard such as ISO14001 (Law & Your Environment, 2024). A standard such as this requires a company to provide evidence of its consideration to the impact it has on the environment this, provides confidence to a potential investor that the business they are about to invest in is focused on long term goals which ensure its sustainability (Nutburn, 2024).

It was for the longevity and environmental sustainability of the business the author wanted to solve a problem which may reduce the consumption of Natural Gas at the refinery hence, a request was made to each of the refineries department managers for information that they may have relating to excessive Natural Gas expenditures. A Technologist responded, informing of a substantial discrepancy of Natural Gas consumption between two seemingly identical Natural Gas furnaces which, had been prominent for a considerable period but, could not be explained. Each of the Natural Gas furnace are known as; Born Heater 1; Born Heater 2 and, were located within the Reduction and Activation step of Kiln Line 1 and Kiln line 2. The descriptive element of both names relative to the manufacturer of the furnace, the identification number relative to the Kiln line of which, it is associated.

1.1. Aims & Objectives

The aim of this report shall be to identify a means to reduce the fixed costs of production by reducing Natural Gas consumption of Born Heater 2 and therefore, reduce impact to the environment. We shall endeavour to complete the following objectives to achieve our aim.

- Familiarisation with the process.
- Research Theory of the process.
- Interview process owner, Team Leaders and Operators.
- Define Project Scope.
- Compile and analyse data.
- Suggest Improvements.

2. Timing Plan

We have used the DMAIC model of Six Sigma to provide a framework for this project therefore, we have used each phase of the acronym; Define, Measure, Analyse, Improve, Control, as milestones for our project time line. It can be seen from our project plan which, has been illustrated in Figure 1 as a Microsoft Excel Gantt Chart, that a milestone is achieved once specific and measurable tasks have been completed. It was important to the author that the timing plan was not restricted to achieving the goals specific to this project but, also included goals associated to their personal, academic and professional circumstances, it can be seen in Figure 1 that time allowances are made for assignment deadlines and, exam preparation. We were mindful to integrate milestones from the Kiln 2 production plan because of an impending outage during which, the process would be isolated for six weeks to allow equipment maintenance.

We have been mindful from the outset that; if potential process improvements were identified, implementing a change to a process which contains hazardous materials would be protracted, therefore, the time allowed for the Improve and Control phases of DMAIC are suggestive rather than applied.

Our original project plan was optimistic with determining durations for milestone, we believe this to be a product of the authors inexperience of the process that they were studying because, as their knowledge grew so too, did the number of potential causations.

On reflection of the projects actual timeline, we concede that delays were incurred because of obstacles which were not considered or, could not have been foreseen. One such deviation was created by the availability of a Hydrogen purity analyser. It was identified during our research phase that there was potential for warm Hydrogen gas which, exited Kiln 2 Born Heater, to become poisoned with Nitrogen this may have had a cooling affect and, may have hindered the effectiveness of the Hydrogen reduction processing of TNOS. After consultation with Vale's Technical team, a planned was drawn to analyse the process gas after the point of potential poisoning however, the analyser which was intended to be used was damaged beyond repair with the lead time for a replacement being longer than this project's deadline. The fluidity of this projects timeline has been a prominent reason why the author has been able to overcome obstacles of this ilk.

[illegible]

3. Research & Literature Survey

3.1. The Process at Clydach

Nickel products are categorised as either Class I or Class II according to the Nickel purity of the product. Class I products have a greater than or equal to 99.8% Nickel content and, at the time of writing can be sold at the London Metal exchange (LME) for \$16230.00 USD per Tonne (LME, 2024). A market exists for a premium product which facilitate bespoke manufacturing process, chemicals used to produce the premium product are confidential because of its substantial markup.

The Mond Process has been used at Clydach to refine raw material into Class I products, since 1902 when it was founded by the German chemist; Ludwig Mond (Vale Europe Ltd., 2024). The refinery has evolved over its many years and has transformed from batch manufacturing to continuous manufacturing which enabled constant chemical processing. The Continuous process has been distributed amongst three plant areas; the Process Gas Plant, primarily used to reform Natural gas into hydrogen; the Process Extraction Plant used to extract Nickel in a gaseous form from the raw material; the Process Manufacturing Plant, used to process gas into the final product which is a raw material for Stainless Steel and Electric Vehicle (EV) batteries manufacturing.

Our topic for this report shall be located within the Process Extraction Plant, an area that has two identical continuous process lines which, operate in parallel by using common inputs and producing a common output that is exported into the Process Manufacturing Plant. The process lines are identified as Kiln Line 1 and Kiln Line 2 of which, each has a series of four kilns; The reduction Kiln; The Activation Kiln; The Volatilisation Kiln; The Stabilisation Kiln, all interconnected with a network of pipework, conveyors, and elevators (Vale Europe Ltd., 2023). The process of each kiln has been divided into two steps; the Reduction and Activation Step, it includes the Reduction and Activation Kiln; the Volatilisation Step includes the Volatilisation and stabilisation Kiln, they are defined so, according to the primary gas which they use; Hydrogen gas in the Reduction & Activation Step; Carbon Monoxide used in the Volatilisation Step.

3.2. The Reduction & Activation Step

Clydach receives 879,000 tonnes of double roasted Nickel Oxide (TNOS) from a refinery in Matsuzaka near Tokyo every year, it's chemical composition comprises of; 75.9% Nickel (Ni); 20.5% Oxygen (O); 1.29% Carbon Monoxide (Co); 0.49% Iron (Fe); 0.13% Copper (Cu); 0.005% Sulphur (S). The material is delivered to the refinery in one tonne bags which, are conveyed into silos where they are stored until required. Demand is determined by the Integrated Business Planner (IBP) who dictates the feed rate at which the process operates, this can vary from 2 to 4 Tonnes per hour (Jones, 2024). When required, the material is discharged from a silo onto belt conveyors which deliver their payload to a storage vessel located on Kiln Line 1 or, a storage vessel located on Kiln 2.

An Additive, Nickel Sulphate Hexahydrate ($NiSO_4 \cdot 6H_2O$), is blended with TNOS before it enters the reduction kiln, this was to introduce Sulphur into the process which, increases the rate of reduction (Oswa, et al., 2008). Bluestone and Umicore manufacture $NiSO_4 \cdot 6H_2O$ for the refinery, having two vendors safeguards the process if, one manufacturer could no longer produce their product (Ebenezer, 2024), each consignment of compound is chemically identically and, delivered with a certificate of conformity. A consignment consists of 22 sealed one tonne bags at a cost of £3430, are delivered to the refinery and stored in a warehouse. At the beginning of shift, a process operator will take eight one tonne bags from the warehouse to; the Nickel Sulphate Preparation Plant where, their content will be transferred to a Link-bin. A Link-bin provides a safe method of dispensing the compound into a storage vessel located on Kiln Line 1 or, a storage vessel located on Kiln Line 2. Whilst,

a Link-bin awaits transfer to a storage vessel, it is held in one of two holding area's; the first, for $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ manufactured by Bluestone the second, manufactured by Umicore. It is believed by Ebenezer (2024), that the purpose of segregation was to ensure that the $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ fed into each kiln remains consistent however, we find contradiction when we review a process data log which, informs of various periods when the $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ manufactured by both vendors have been used on one kiln line (Vale Europe Ltd., 2024).

Each material stored in their associated storage vessels located on Kiln Line 2 are discharged into weigh hoppers which create batches of TNOS and $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ in accordance to weight. The TNOS weigh hopper discharges into a rotary valve which feeds into a rotary screw conveyor, the speed of both are variable and controlled by a Variable Speed Drive (VSD), this is programmed to alter the speed of the rotary valve and conveyor in accordance with an hourly feed rate the process operator decides to select. Likewise, the $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ weigh hopper discharges into a rotary valve and rotary screw conveyor, the speed of which is controlled by a VSD that is unrelated to the aforementioned VSD but, is also controlled by the process operator selecting an appropriate hourly feed rate. When the $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ reaches the end of the rotary screw conveyor, the materials drops into a discharge pipe which enters into the top of the TNOS rotary screw conveyor where, both materials are blended together.

At the end of the TNOS rotary screw conveyer the blended material falls into the boot of a bucket elevator then, lifted up to the top of the process where, it is allowed to fall through various seal hoppers that have been designed to prevent the escape of Hydrogen gas as the material moves closer to the Reduction Kiln. Once inside the Reduction Kiln, the material is conveyed through the 35m long rotating vessel at a speed dictated by the kilns rotational speed and it's 3° horizontal decline. The kiln's Internal mild steel scoops agitate the material, exposing as much of its surface area to the warm Hydrogen gas during the 48 hour incubation period. The reduced material leaves the Reduction Kiln via a rotary valve and drops into the Activation Kiln.

Whilst in the Reduction Kiln, the blended material is enveloped by Hydrogen which flows through the 4m diameter kiln in the opposite direction to the flow of material then, exits via an outlet located near the inlet of which the blended material enters. The Hydrogen gas is heated as it flows through a Born Heater which, has a radiant zone where four stainless steel tubes, each with a diameter of 100mm which, provide a conduit for Hydrogen gas to flow. The tubes pass through the radiant zone of the Born Heater seven times, allowing the gas within sufficient time to be heated. Three combustion burners provide heat to the Radiant Zone by burning Natural Gas in the presence of a naturally induced flow of air which enters at the bottom of the combustion chamber. The temperature is set by a Process Operator in a Digital Control System (DCS) which can range of 420°C and 530°C, this provides a set-point for instrumentation embedded within a closed-loop control system to achieve and maintain. If the temperature is not maintained a thermocoupler will detect the deviating process variable and, inform the DCS of the error, a controller would adjust a 4-20 mA signal to a 'Current to Pressure' converter, applying a proportional 3-15 psi response of instrument air to a valve positioner, which would adjust the amount of Natural Gas flowing into the combustion units of the three burners ergo, controlling the temperature of Hydrogen Gas entering the process.

When the blended material enters the Activation Kiln, a rotating vessel that has a length of 20m and a diameter of 2m, it moves in the same manner as it does in the Reduction Kiln but, has a shorter incubation period of 8 hours (Bevan, 2024). During its transition through the kiln the material is again agitated and exposed to a flow of Sulphur Dioxide (SO_2) and Hydrogen which both flow in the same direction that the material is travelling in. This, according to Vale Europe Ltd. (2023), produces a

Sulphide within the material which enhances Nickel extraction in the following Volatilisation Step however, Vale Europe Ltd. (2023), confesses that the exact mechanism of this reaction is unknown.

We can see in Figure 2, a block diagram that identifies the necessary inputs and, corresponding by-products, of the Reduction and Activation step, Figure 3 simplifies further by illustrating the main process flow, excluding inputs and outputs associated with sub-processes. The product of this process is a material that has a Nickel content greater than 89% which, is then able to be processed in the following Volatilization step.

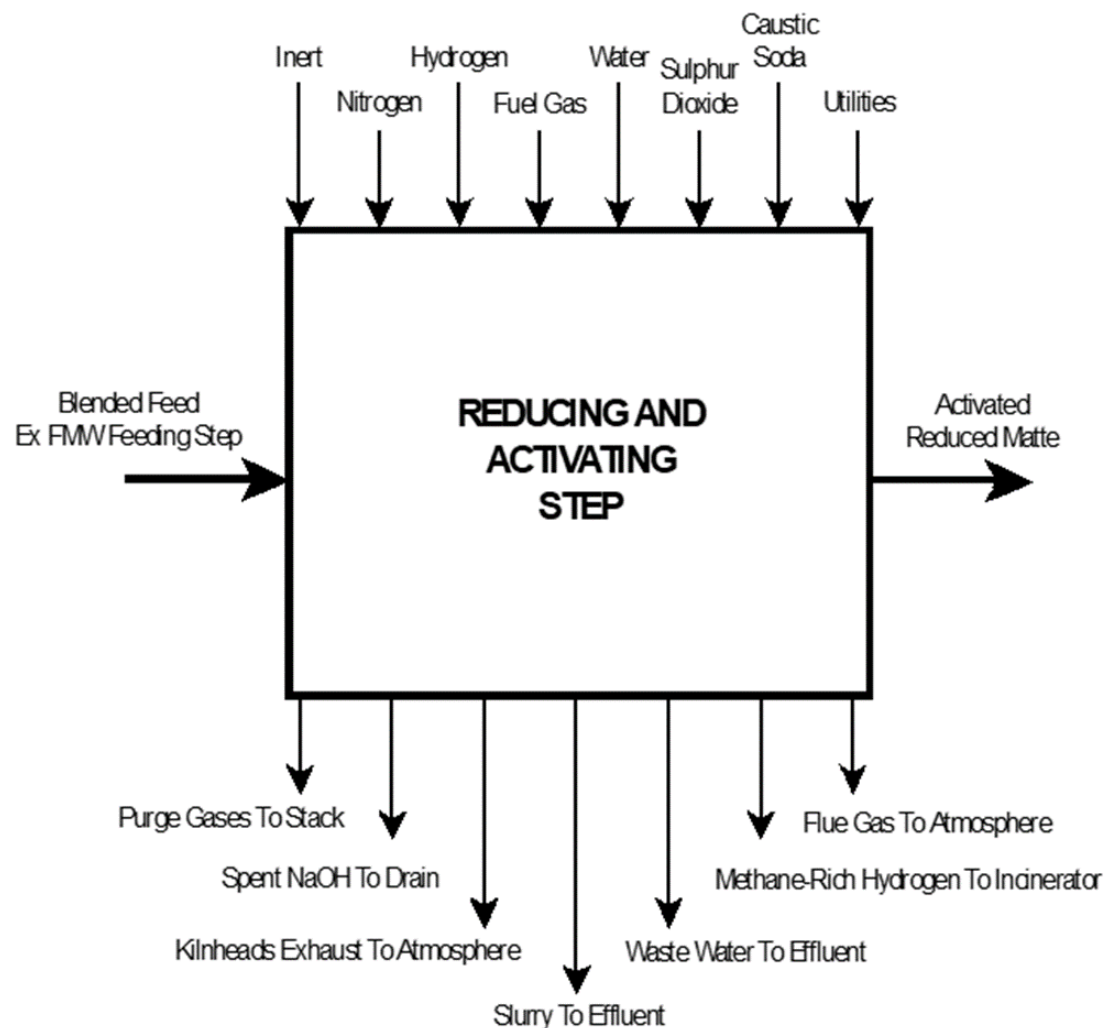


Figure 2, Inputs and outputs of Reduction and Activation Step (Vale Europe Ltd., 2023)

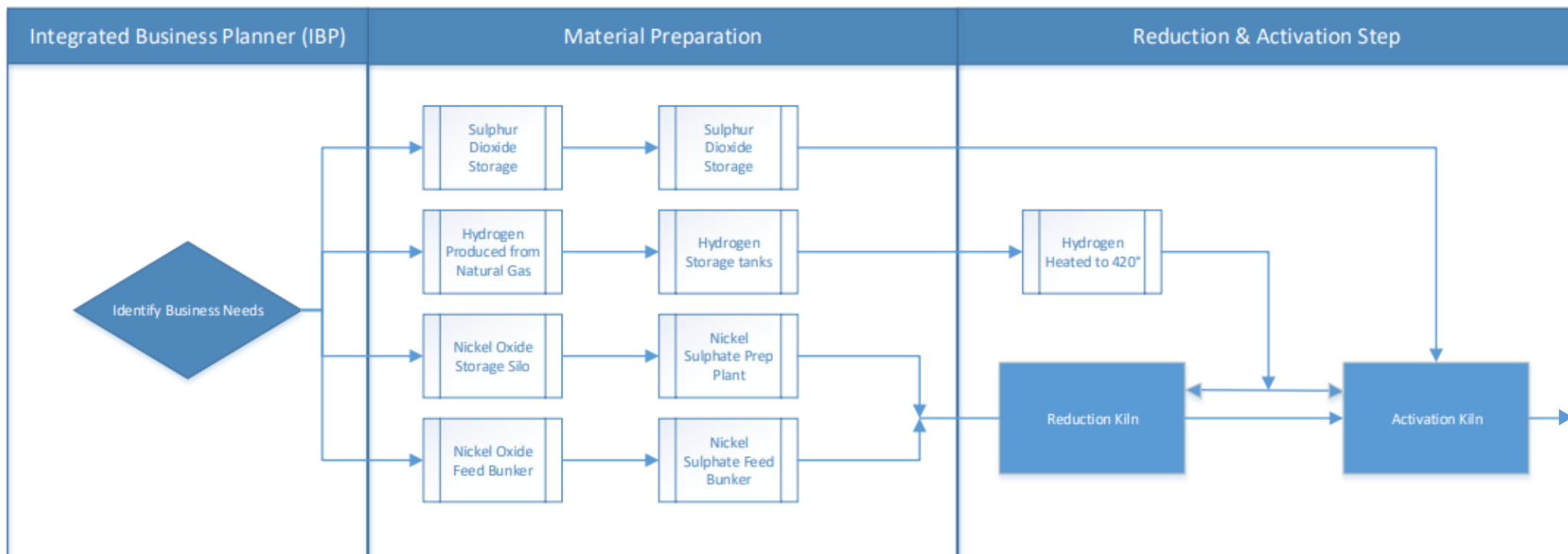


Figure 3, Process Flow up to and, including the Reduction & Activation Step

3.3. Theory of the Reduction Process

The Reduction Kiln's function is to remove Oxygen from Nickel Oxide, it achieves this by using the Hydrogen Reduction Process (REDOX), an established method that provokes a reaction between Oxygen atoms embedded in the lattice structure of a metal oxide molecule and, the atoms of heated Hydrogen gas. The result of the reaction is the formation of gaseous water molecules which, are carried away in the direction of flowing Hydrogen. Hydrogen gas is used for its notable characteristics as a reducing agent because it is eager to shed its only electron that orbits in its most outer shell so, that the element may become a noble gas (Science Notes, 2024). Hydrogen is adsorbed onto the surface of the NiO molecule where its electron is transferred to Oxygen at the solid-gas interface causing the transformed Hydrogen proton to have an electropositive charge this, creates an amenable condition allowing a polar covalent bond to form between a pair of electrons from an Oxygen atom which, has an electronegative charge that would be created by receiving the Hydrogen atoms disassociated electron (Brown, 2015). A Hydrogen atom has two vacancies in its valence shell that allows for a bond to be formed with another Oxygen atom, this reaction forms a water (H_2O) molecule (Averill & Eldredge, 2012).

Affective Migration of Oxygen atoms from the TNOS compound is subject to; type and concentration of the reduction gas used, reaction temperature, time exposed to the reduction gas, porosity of the particle and surface area (Plascenia & Utigard, 2009), furthermore, Lee & min (2019), find correlation between smaller particle size and a decrease in the required reduction temperature.

Quality of the Reduction process is determined by the Nickel versus Nickel Oxide ratio of a processed particle and, a processed particle's Specific Surface Area (SSA). The Nickel versus Nickel Oxide ratio strives to achieve a less than 2% Nickel Oxide (Unreduced) value, this ensures the material introduced to the Volatilisation step is of a high Nickel content and, is able to be processed which, mitigates processing costs that do not produce a yield. A high SSA value enables a high Nickel extraction rate from a Nickel particle whilst in the Volatilisation step (Vale Europe Ltd., 2023) therefore, maximising production yield.

Producing a quality product during the Hydrogen reduction process requires the correct process conditions and chemical components for the necessary reactions to take place. The work by Moore (2015), on effects of the addition of $NiSO_4 \cdot 6H_2O$ to Nickel Oxide prior to the material entering the process, explains that there are variables which need to be considered to ensure that unreduced Nickel Oxide is limited and, that SSA are maximised. He informs us of the following variables which need to be considered; ratio of $NiSO_4 \cdot 6H_2O$ to TNOS; impurities in both $NiSO_4 \cdot 6H_2O$ and TNOS; temperature of process; duration exposed to process; Hydrogen flow rate; purity of Hydrogen, Lascelles (2004), investigation into an alternative source of nickel sulphate for the kiln plant, also recognised that the addition of $NiSO_4 \cdot 6H_2O$ caused modification to the pore structure of the Nickel molecule because of the higher rate of reduction (Lascelles, 1986), this positively influences Nickel extraction rates in the Volatilisation Step due to the increase of specific surface area. $NiSO_4 \cdot 6H_2O$ facilitate migration of Oxygen atoms from Nickel molecules by coating a layer of Sulphur onto the surface of a Nickel Oxide molecule. To do so, $NiSO_4 \cdot 6H_2O$ is decomposed at a temperature greater than $100^\circ C$ which causes the ionic bonds of the compound to rupture allowing the required sulphur to be liberated (National Library of Medicine, 2024).

It is the consensus of Foran (2014) and Moore (2015), that the ideal ratio of Nickel Sulphate Hexahydrate should introduce a 0.4% proportion of Sulphur for the total combined mass of the blended feed that is to be processed, at this ratio they conclude that reduction temperature, residence time, and the probability of material sintering shall be reduced.

3.4. Theory of the Activation Process

As aforementioned, Vale Europe Ltd. (2023), knows little of the reaction between Ni, H₂, and SO₂ which happens in the Activation Kiln but, they state with a large degree of certainty what they believe to be the product of the following reaction.

“Nickel + Sulphur Dioxide + Hydrogen = Sulphided Nickel + Water” (Vale Europe Ltd., 2023)

A paper investigating the recovery of SO₂ in the atmosphere, provides education of how this reaction may occur by informing that the Nickel particals in the material may act as a catalyst. According to Lu, et al. (2021), Nickel Phosphate is a catalyst for the hydrogenation of SO₂ creating Hydrogen Sulfide (H₂S), and based on the work by Zhang, et al. (2019), exposing pure Nickel to H₂S will lead to the poisoning of Nickel with Sulphur resulting in a Sulphided Nickel product. If using a Nickel based catalyst to hydrogenate SO₂, efficiency of the process will depend on the morphology of the catalyst and the temperature of which the process has been exposed to, Lu, et al. (2021), says that this should be in the range of 200°C - 300°C, however they also report that a metal-free phosphate mesoporous silica catalyst can be used to improve performance if temperature is below 200°C. Sulphur poisoning of pure Nickel takes place when H₂S molecules are adsorbed onto active sites on the surface of Nickel partical with a weak bond forming between a Sulphur atom and the Nickel atoms. The activation energy of bonds are greater than that of the bonds between a Sulphur atom and the Hydrogen atoms of a H₂S molecule resulting in their fracture and the decomposition of the H₂S molecule (Zhang, et al., 2019).

It is assumed that the intention of the process is to prevent liberated Oxygen atoms, a by-product of the formation of H₂S, from re-oxidising the pure Nickel product by bonding them with Hydrogen atoms recently liberated from H₂S molecules to form water vapour which can be removed from the process in a condensation column external to the Activation Kiln. For this to be effective the ambient temperature, which will provide the activation energy needs to be above 300°C (Jolly, 2024).

3.5. Volatilisation Step

The processed material leaving the Activation Kiln enters the Volatilisation Kiln, a 55m long rotating vessel that has a horizontal decline of 3°, its Internal mild steel scoops agitate the material, exposing the material particles to Carbon Monoxide (CO). The CO flows through the 4.6m diameter kiln in the opposing direction to the flow of the material, transforming into Nickel Carbonyl gas then, exiting through a gas outlet pipe near to the point of which the material entered the kiln. The material is incubated in the kiln for 12 days (D'Angeli, 2024), after which it exits through a rotary valve and conveyed into the Stabilisation Kiln, once adequately cooled, the residual material is sent to a precious metals refinery where metals such as Cobalt and Iron are extracted.

Nickel Carbonyl gas which exits the Volatilisation Kiln flows through a circuit of pipework known as the Carbonyl Circuit. The Carbonyl circuit transports gas from the Process Extraction Plant to the Process Manufacturing Plant where it is decomposed into the final product. The decomposing process, breaks bonds between CO and Ni atoms allowing CO to flow back to the Process Extraction Plant where it is recycled by being introduced back into the Volatilisation Kiln. It can be seen in Figure 4, a block diagram that identifies the necessary inputs and, corresponding outputs required to produce an adequate product in the Volatilization Step.

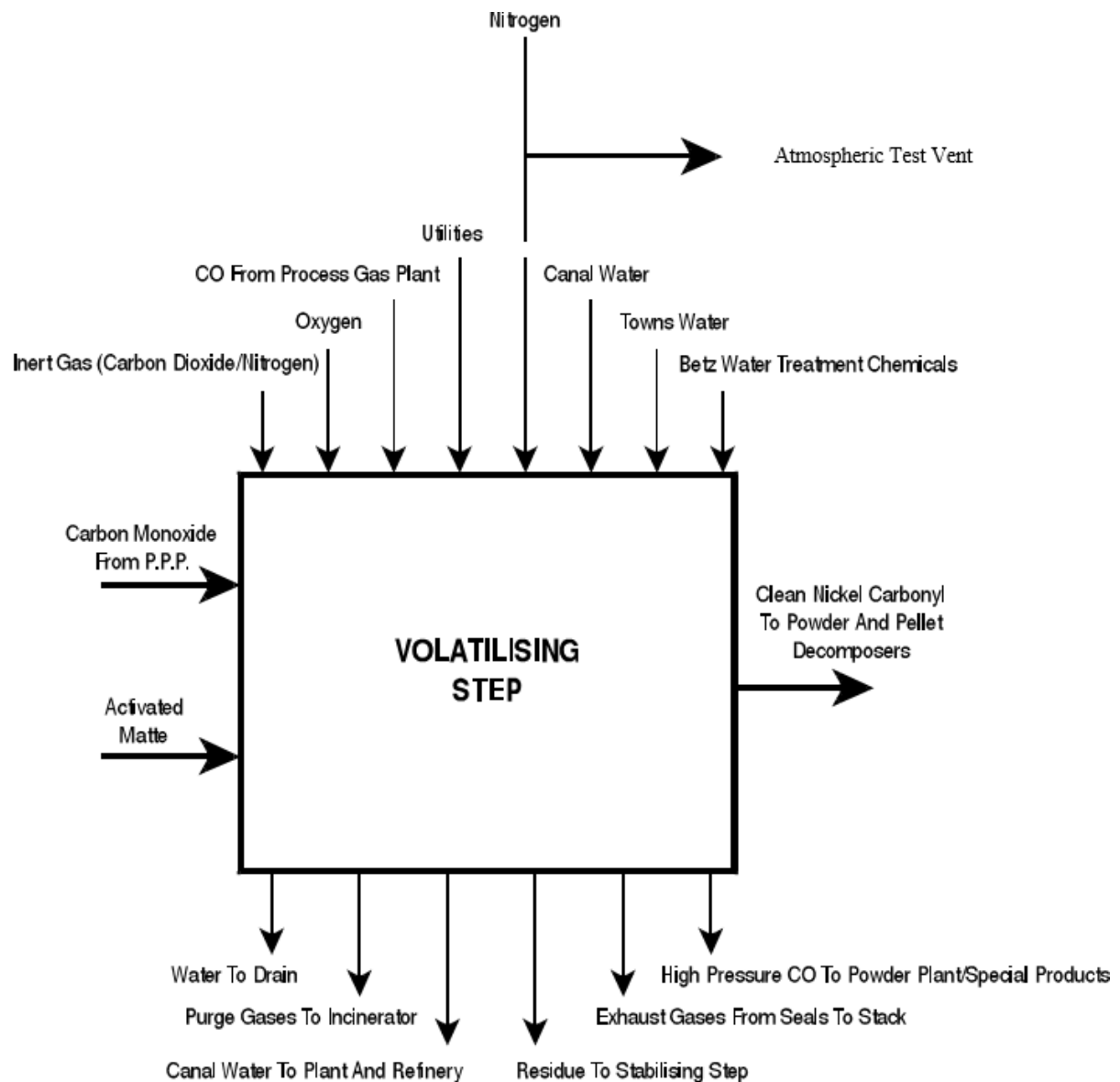


Figure 4, Inputs and outputs of the Volatilisation Step (Vale Europe Ltd., 2024)

3.6. Theory of the Volatilisation Step

The Activated material enters the volatilisation kiln with an ideal Sulphur content of 1.7%, which has permeated multiple layers of the Nickel molecule, by doing so has caused the covalent bonds holding the molecules atoms together to weaken (Greig, 1972). As the material moves through the Volatilisation Kiln, at a temperature between a range of 50 to 60°C (Lewis, 2023), a Carbon atom of the CO molecule forms a bond with a Nickel atom, it's energy severs the bonds between other atoms, creating a gases carbonyl molecule called Nickel tetracarbonyl gas (Ni_4). Once an atom has been pulled away from the surface, another Nickel atom is exposed and, if part of the sulphated layer, allows for the process to be repeated. The efficiency of this process is reviewed by measuring the ratio of Ni_4 to CO, the target ratio should be greater than or equal to 90%, according to Poland (2023), an influencer of this metric is the Specific Surface Area (SSA) of the reduced and activated marterial.

3.7. Problem Statement

A measurement of Natural Gas consumed by each Born Heater was recorded every week during a period in 2023, the measurement was relative to the amount TNOS processed by each of the

Reduction and Activation Steps. The graph illustrated in Figure 5 shows measurements recorded for Kiln 1 while, Figure 6 illustrates measurements for Kiln 2, each green plot represents a measurement of gas consumption (Y-axis) V's the quantity of TNOS processed in the Reduction and Activation step (X-axis). An orange plot can be seen on each graph, this represents a measurement taken for Week 45 of 2023, a comparison of the this measurement shows that for approximately the same quantity of TNOS processed (680 Tonnes), gas consumption for Born Heater 2 was significantly higher than that of Born Heater 2 - 320,000KW/h and 275,000KW/h respectively. The monitory comparison was calculated with the actual cost paid for each KW/h which was £0.0549 (Sterling, 2023), the cost of gas consumption for Born Heater 1 was £15,097.50 and the cost for Born Heater 2 was £17,568, a total loss of £128,466 can be calculated for the inefficiency of Born Heater 2 over a 52 week period.

An interview with the Process Superintendent provided the problem statement which we will attempt to solve; "The Hydrogen gas exiting Kiln 2 Born Heater needs to be hotter than that of the gas exiting Kiln 1 Born Heater, this helps us to control the percentage of Nickel Oxide that has not been reduced and, to increase the Specific Surface Area of the material....." (Poland, 2023). This is confirmed by reviewing the Distributed Control System (DCS), which provides a recording of process variables, we can see from Figure 7, a comparison of the desired temperature (Set-Point), for the Hydrogen gas exiting Kiln 1 & Kiln 2 Born Heaters. The period reviewed is for quarter 4 of 2023 and, shows that Kiln 2 (Orange plot), has a consistently higher setpoint than that of Kiln 1 (Blue plot). Furthermore, we find additional confirmation when we compare the percentage of Nickel Oxide that was not reduced by Kiln 2 with the setpoint for the desired temperature of Hydrogen, it can be seen in Figure 8 that, as the percentage of Nickel not reduced climbs past the upper limit of 2% the setpoint is increased.

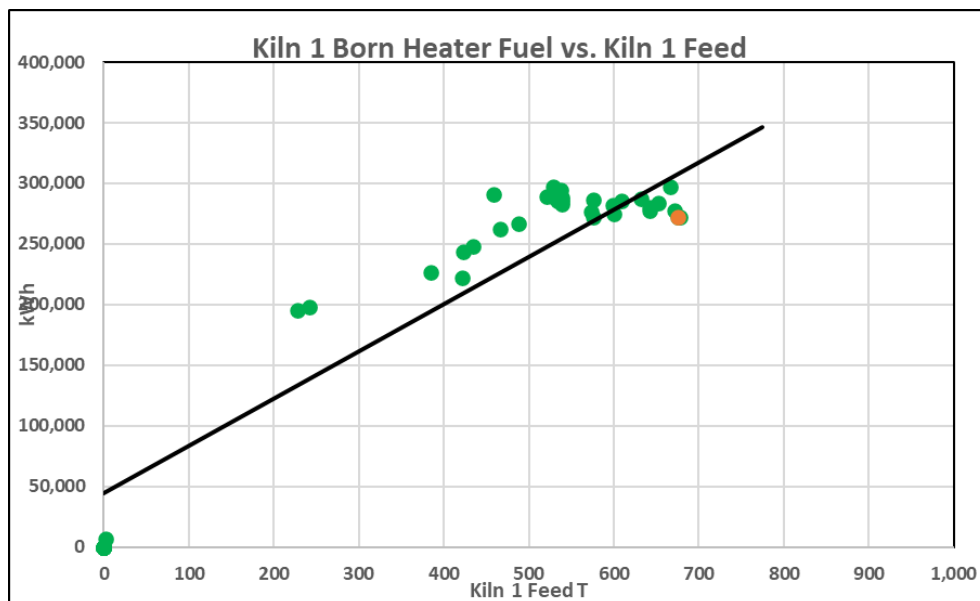


Figure 5, Trend of Natural Gas consumption relative to quantity of TNOS processed through Kiln Line 1 (Sterling, 2023).

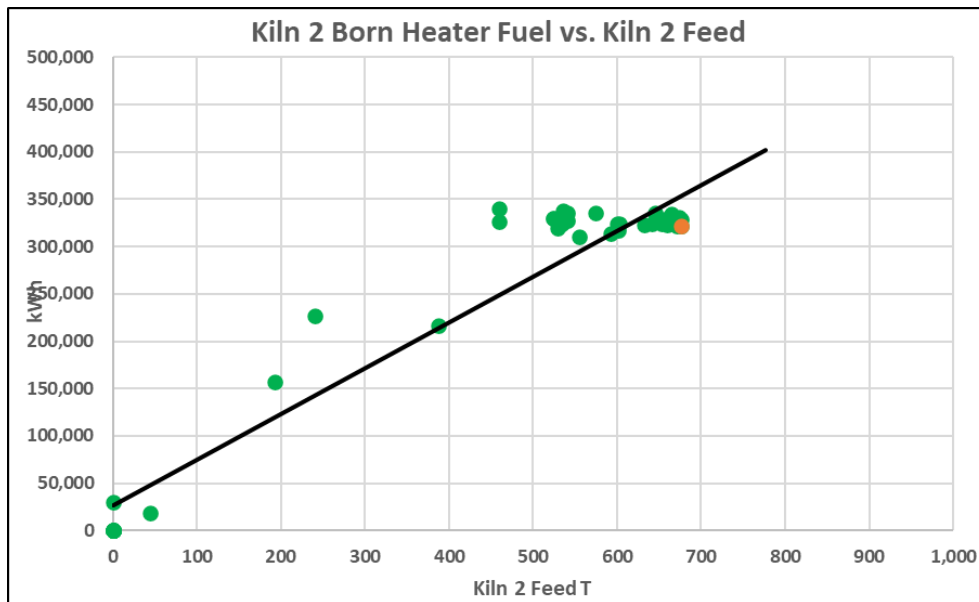


Figure 6, Trend of Natural Gas consumption relative to quantity of TNOS processed through Kiln Line 2 (Sterling, 2023).

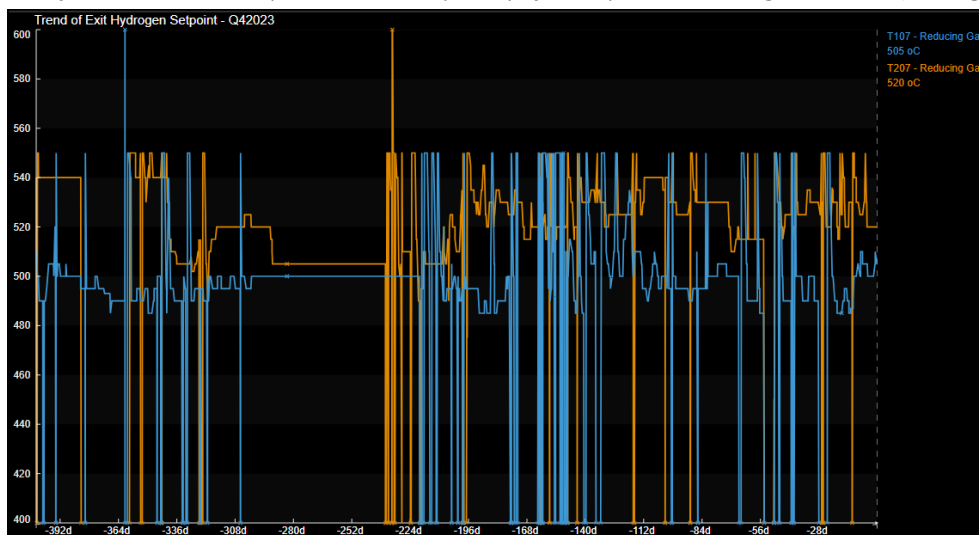


Figure 7, Comparison between Kiln 1 and Kiln 2 Born Heater's Exit Hydrogen Temperature Setpoint (Foxboro DSC, 2024)

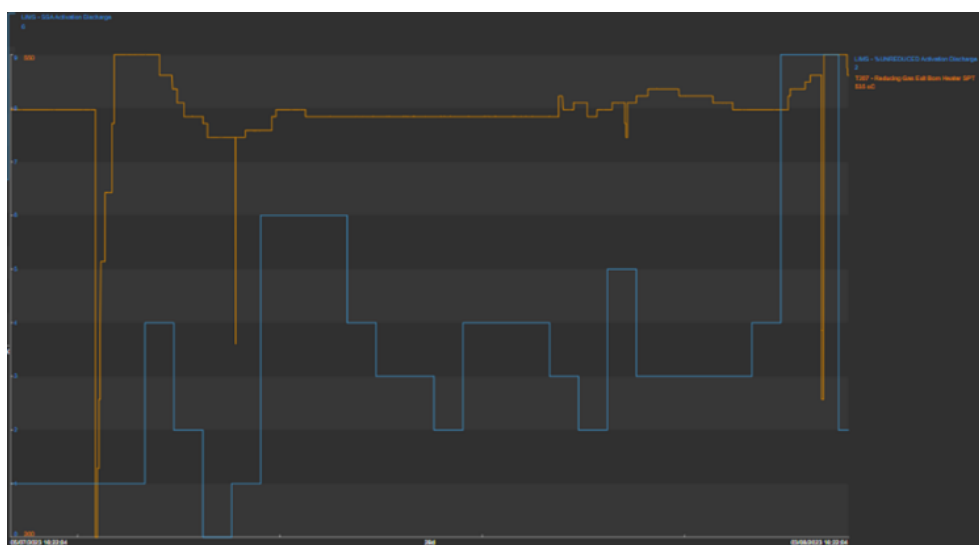


Figure 8, Comparison between Kiln 2 Hydrogen temperature setpoint and, Kiln 2 %Unreduced value (Foxboro DSC, 2024).

3.8. Previous Investigations

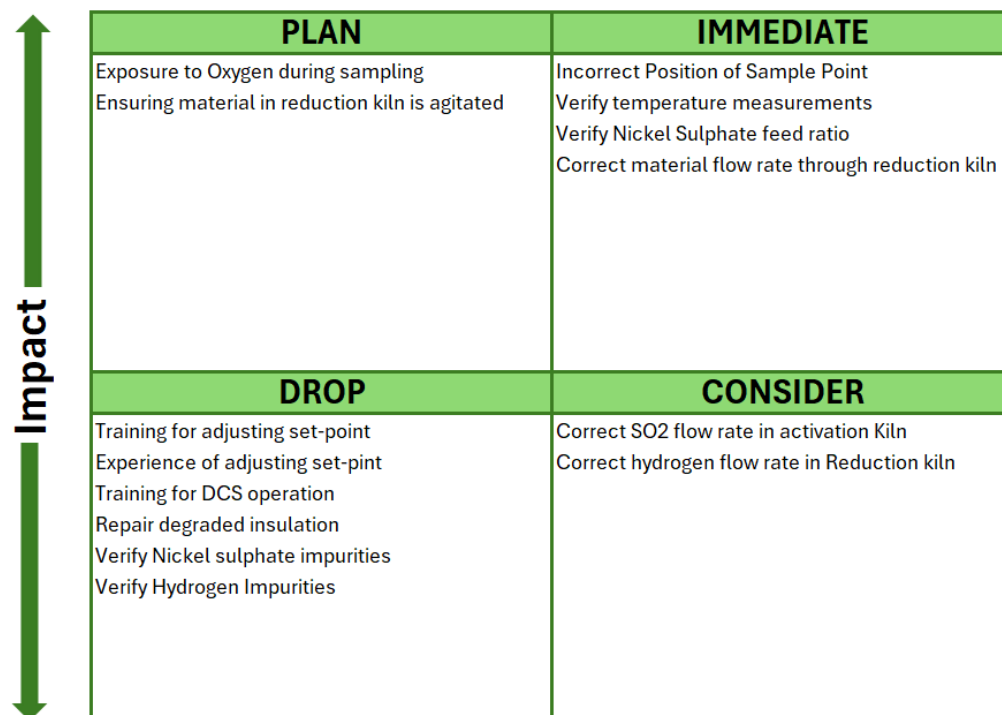
The discrepancy of gas consumption for each Born Heater has been evident for many years, gas measurements identify the issue as far back as 2004 (Vale Europe Ltd., 2024) prompting a report to be commissioned by the refineries leadership team in 2013.

The report conducted by CR Plus Engineering (Appendix A), focused on the combustion inefficiencies of each Born Heater, concluding that Kiln 2 Born Heater was less efficient than that of Kiln 1 because of the accumulation of additional heat losses from the Waste Gas Stack and exposed supporting structure. The report proceeds to suggest a “quick Win” option that specifies the remedial work to worn seals and insulation at a cost of £20,000 and, a “long term” option which requires a full replacement of the Born Heater at a cost of £950,000 (CR Plus Ltd, 2013). It was obvious to the reader that the aforementioned study did not consider the Born Heater as a component of a system, rather, an isolated asset with assumptions made of the characteristics for the remainder of the system therefore, there was little contemplation of the external influences that may impact the efficiency of the Born Heater.

4. Investigation

Interviews were conducted to establish the; Voice of the Customer and, the Voice of the Process, in doing so the entire system, that is; the Reduction and Activation Step was considered. A Cause and Effect diagram (Figure 10), was created to document relevant influences which, may contribute to this reports problem statement. Establishing potential improvement for a system as complex and, as hazardous as this shall require considerable support from a team of process experts which, depending on the enhancement could take an extensive period of time, it is for this reason, we have used an Impact Matrix (Figure 9), and have establish where our time will be best spent.

After consideration and consultation with stakeholders we decided to pursue the “verify the Nickel Sulphate Hexahydrate feed ratio” cause. We then focused on finding a method to measure the relationship between the process input and process output, we analysed data recorded in the Bonham data log finding a correlation between the use of $NiSO_4 \cdot 6H_2O$ from various manufacturers and, the percentage of unreduced Nickel Oxide. The relationship has been illustrated as a combined bar and line graph in Figure 11, bars rising above the horizontal axis identify a period when $NiSO_4 \cdot 6H_2O$ manufactured by Umicore was used by Kiln Line 2, the bars dropping below the horizontal axis show the period when the compound was manufactured by Bluestone. The blue line plot represents the measurement of percentage unreduced Nickel Oxide during the period that both manufacturers of the compound was used, it has a negative offset of 72 hours which accounts for the time required for the blend of TNOS and $NiSO_4 \cdot 6H_2O$ to be processed by the reduction kiln and, the activation kiln where after, a sample was taken every 24 hours to analyse percentage unreduced Nickel Oxide. We can see from the dashed plot of the linear trend that; the percentage unreduced Nickel Oxide value decreases when the $NiSO_4 \cdot 6H_2O$ component of the blended feed was manufactured by Bluestone, when we numerically analyse the data for an equal period either side of the date the manufacturer of the compound was changed, it was found that the average percentage of unreduced Nickel during a campaign of Umicore was 3.4%, in contrast a campaign of Bluestone was 1.15%.



Impact  	PLAN	IMMEDIATE
	Exposure to Oxygen during sampling Ensuring material in reduction kiln is agitated	Incorrect Position of Sample Point Verify temperature measurements Verify Nickel Sulphate feed ratio Correct material flow rate through reduction kiln
	DROP	CONSIDER
	Training for adjusting set-point Experience of adjusting set-pint Training for DCS operation Repair degraded insulation Verify Nickel sulphate impurities Verify Hydrogen Impurities	Correct SO2 flow rate in activation Kiln Correct hydrogen flow rate in Reduction kiln

Figure 9, Impact Matrix evaluating causes of the problem statement (Croft, 2024)

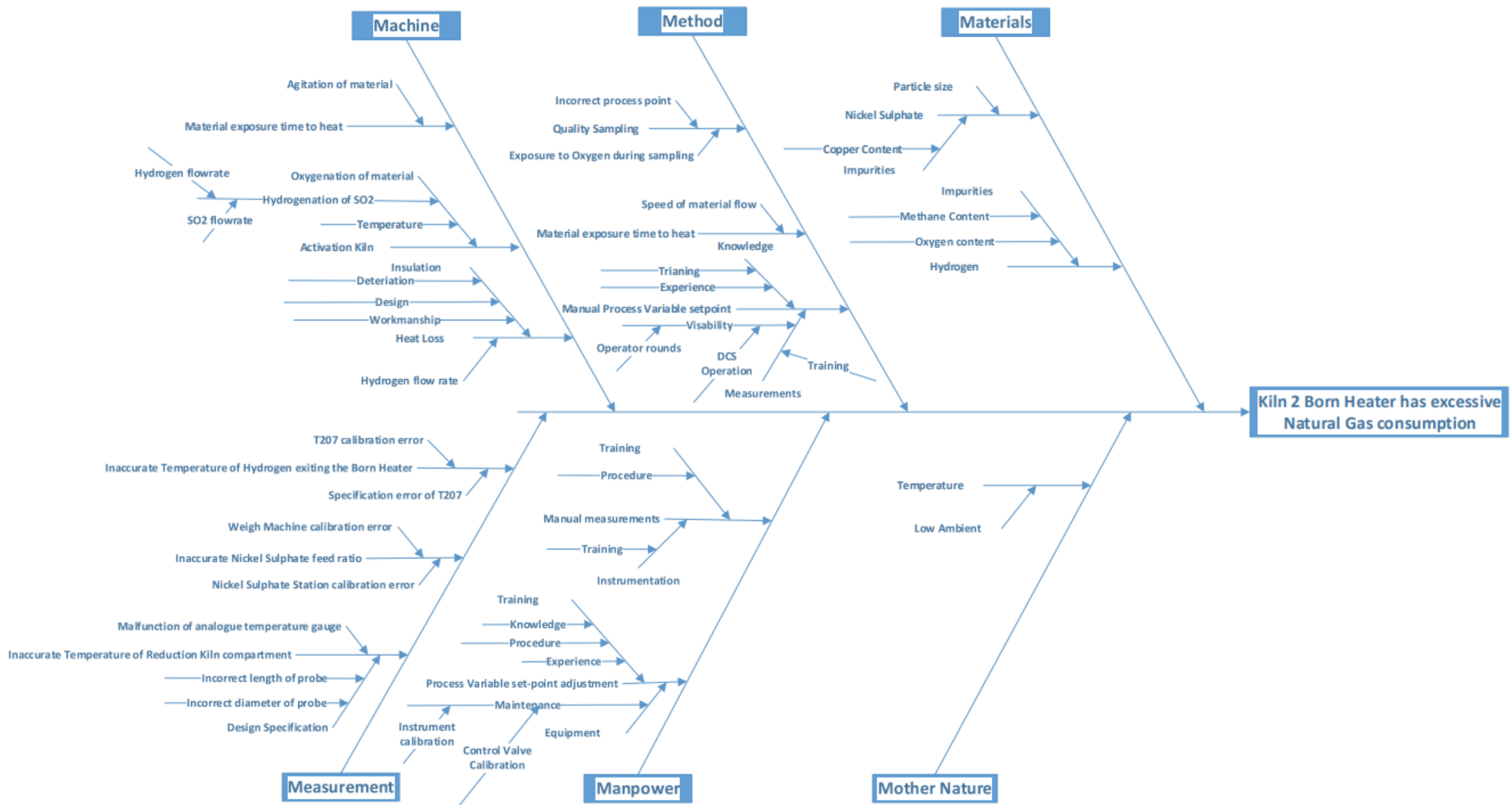


Figure 10, Cause and Effect diagram used to establish potential reasons why Kiln 2 Born heater consumes additional Natural Gas

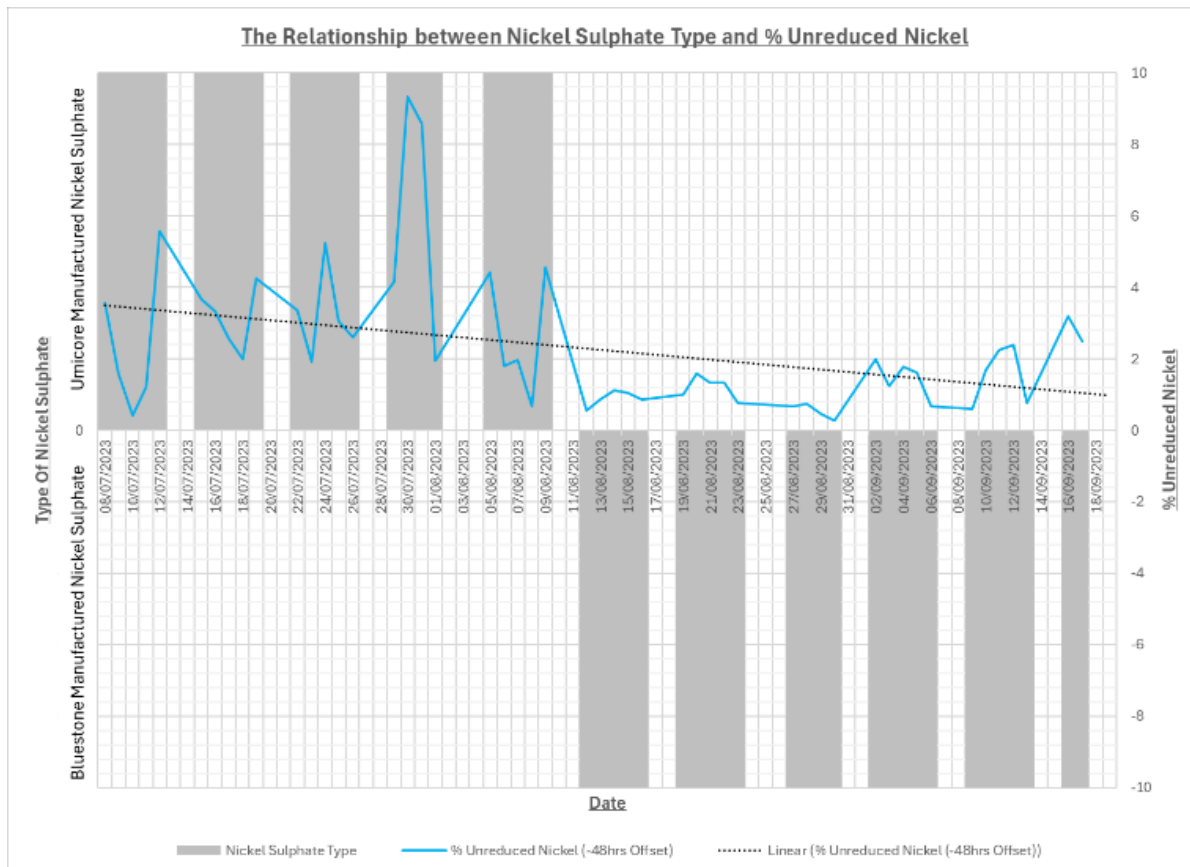


Figure 11, The Relationship between Nickel Sulphate Type and % Unreduced Nickel

We proceed to gather the information we have learnt to establish reasons why a different manufacturer of this compound would have a consequence of increasing the temperature set-point of the Hydrogen gas exiting Kiln 2 Born Heater , we employ the 5-Whys exercise (shown in Figure 12)

Problem Statement Kiln 2 Born Heater requires more Natural Gas compared to Kiln 1 Born Heater.				
1st Why	2nd Why	3rd Why	4th Why	5th Why
Temperature of born heater is controlled by T207 which has a higher setpoint than Kiln 1.	The Internal temperature at the centre of the reduction kiln 2 has an impact on the purity of Nickel extracted from the nickel oxide material.	The Percentage of Unreduced material is greater than 2%	Properties of nickel sulphate hexahydrate recived from different manufacturuer are different from previous manufacturer.	- Lower Sulphur content - Change in mass - Larger particle size - Higher Impurities

Figure 12, 5-Whys exercise to identify root causes

We shall perform sampling and analysis to determine the actual content of Sulphur contained in a mass unit of $NiSO_4 \cdot 6H_2O$ and, to find the sample's pH value, in doing so, we aim to find evidence which may support a root cause. A sample of $NiSO_4 \cdot 6H_2O$ shall be taken from four sealed bags of the compound prior to its introduction to the process. The compound is a cancerogenic therefore, the sample was collected by a process operator who donned the appropriate Personal Protective Equipment, on tearing the sealed bag they commented on the odour released. A measurement of

250ml of compound was taken from 2 bags which were supplied by the Bluestone vendor and, 2 bags which were supplied by the Umicore vendor. Each sample was labelled with details of the name of sampler; date sample taken; manufacturer; lot/batch number. To perform analysis that identify Sulphur content, a Quality Assurance Laboratory technicians dissolved 0.5g +/- 0.0005g of $NiSO_4 \cdot 6H_2O$ in to a 100ml flat bottom boiling glass. 12ml of Perchloric acid and 3 ml of concentrated nitric acid were added to the boiling glass which, was then placed on a hotplate for 5 minutes to begin the dissolution process. Once the sample had dissolved the glass was removed from the hot plate and left to cool, at room temperature anti-bumping granules, 40ml of deionised water and, 20ml of Zinc standard solution was added. Plastic sample holders were used to prepare the sample for inserting into the analyser, each have three parts: an outer ring, an inner ring and a cap, these are assembled in a fashion which prevents a liquid sample from leaking while analysed. The samples were loaded into a X-ray Fluorescence Spectrometry, an instrument which emits a primary source of radiation that is used to detect various elements within a sample by analysing the secondary radiation emitted from the material, by doing so, the radiation absorbed by the material can be calculated and, the material identified (Nasrazadani & Hassani, 2016).

Analysis for pH saw a further 40g +/-0.1g of each sample weighed and transferred to a 100ml flat bottom boiling flask where deionised water was added. A Mettler Toledo Expert Pro Electrode was used in conjunction with the Mettler Toledo seven Easy pH analyser so that an accurate pH measurement could be established. The electrode was introduced, one at a time, to three known standard solution; pH7, pH4 and pH10, these were used to verify the analysers calibration, after each verification test the electrode was rinsed with deionised water to eliminate contamination. The electrode was dipped into a sample of dissolved Nickel sulphate hexahydrate, the solution was agitated with a magnetic stirring bar and left until the analysers digital reading stabilised at which point the measurement was recorded. The Value of a pH measurement can range from 0 to 14, where 0 is very acidic, 7 is neutral and, 14 is very alkaline. We anticipate that our results will equal to or, greater than 5 if not, Laker (2008) advises that the only consequence is that the internal steelwork associated with the reduction and activation step will be subjected to an increase in corrosion, Roselina, et al., (2013) contradicts Laker (2008) by informing of an additional consequence, they suggest that a variation in pH can have an effect on the structure of a Nickel molecule.

Our expectation was for the content of Sulphur to be consistent with the value obtained from the analytical method used to find the atomic mass of the element for 1 molecule of $NiSO_4 \cdot 6H_2O$, we proceeded by using the molecular formula of the compound to identify the number of atoms of each element, we multiplied the total of atoms of an element by each elements atomic mass weight to find the total mass of an element and, have shown our results in Table 1 (Chemical Aid, 2024).

Nickel Sulphate Hexahydrate has a mass weight of 262.87 g/mole (Kathiravan, et al., 2016) therefore, it can determine the proportion of Sulphur contained within a mass unit of the compound shall be 12.198%. It is important that our experiment validates this percentage because it has been used in Equation 1 to produce a guide (Figure 14), for an Process Operator when they adjust the feed streams for TNOS and $NiSO_4 \cdot 6H_2O$, if Sulphur content is below that of which found by the analytical method, the operator would unknowingly starve the Hydrogen Reduction process of the Sulphur it needs to be effective. We reviewed certification received from vendors upon delivery for validation of Sulphur quantity however, found that this is not stated instead the Nickel content of the compound was specified as 22.296% (Appendix C), from this we use the proportion of Nickel identified by the analytical method which, has been presented in Table 1, for generating Equation 2 which, provides validation of the correct amount of Nickel is present but, does not confirm that the element is part of the desired compound, it could be speculated that the element exists as Nickel Oxide in which case,

would be considered as an impurity because the mass unit weight of the compound would change diluting the quantity of Sulphur contained in the lot.

We elaborate with the following example, consider a delivery having a volume of 1m^3 and, on the basis of the delivery being 100% Nickel Sulphate Hexahydrate would have a mass of 3680 kg of which, 448.89 kg would be Sulphur but, when an alternative scenario is considered whereby, a delivery consists of; 90% Nickel Sulphate Hexahydrate that has a density of 3.68 g/cm^3 (National Center for Biotechnology Information, 2024); 10% Nickel that has a density of 8.9 g/cm^3 (National Center for Biotechnology Information, 2024), Equation 3 shows that a consignment of a mass 3680 kg would have an overall reduced of Sulphur content in the lot to 404 kg, for context; this would reduce the Sulphur component of the total mass of product fed into the Hydrogen reduction process from 0.488% to 0.439% (Equation 1).

Each delivery of Nickel Sulphate was accompanied with certification that was issues by the manufacturer to verify quality and weight of their product (Appendix C, Appendix D, Appendix E, Appendix F), we make comparisons to our experimental results for validation that quality has not been compromised during transportation.

Table 1, Total mass weight of each element contained in Nickel Sulphate Hexahydrate (Chemical Aid, 2024)

Element	Number of Atoms	Molar Mass (g/mol)	Total Molar Mass (g/mol)
Nickel (Ni)	1	58.6934	58.6934
Sulphur (S)	1	32.065	32.065
Oxygen (O)	10	15.9994	159.994
Hydrogen (H)	12	1.00794	12.09528

TOTAL HOURLY FEED RATE (KG / HR)	25 : 1 FMW TO NiSO ₄ RATIO		30 : 1 FMW TO NiSO ₄ RATIO		35 : 1 FMW TO NiSO ₄ RATIO	
	HOURLY FMW FEED RATE (KG / HR)	HOURLY NiSO ₄ FEED RATE (KG / HR)	HOURLY FMW FEED RATE (KG / HR)	HOURLY NiSO ₄ FEED RATE (KG / HR)	HOURLY FMW FEED RATE (KG / HR)	HOURLY NiSO ₄ FEED RATE (KG / HR)
2,000	1,920	80	1,933	67	1,943	57
2,100	2,016	84	2,030	70	2,040	60
2,200	2,112	88	2,127	73	2,137	63
2,300	2,208	92	2,223	77	2,234	66
2,400	2,304	96	2,320	80	2,331	69
2,500	2,400	100	2,417	83	2,429	71
2,600	2,496	104	2,513	87	2,526	74
2,700	2,592	108	2,610	90	2,623	77
2,800	2,688	112	2,707	93	2,720	80
2,900	2,784	116	2,803	97	2,817	83
3,000	2,880	120	2,900	100	2,914	86
3,100	2,976	124	2,997	103	3,011	89
3,200	3,072	128	3,093	107	3,109	91
3,300	3,168	132	3,190	110	3,206	94

Figure 13, Photograph of rate parameters for a 25:1 feed ratio for a total feed rate between 2,000 and 3,300 kg per hour.

TOTAL HOURLY FEED RATE (KG / HR)	25 : 1 FMW TO NiSO ₄ RATIO		30 : 1 FMW TO NiSO ₄ RATIO		35 : 1 FMW TO NiSO ₄ RATIO	
	HOURLY FMW	HOURLY NiSO ₄	HOURLY FMW	HOURLY NiSO ₄	HOURLY FMW	HOURLY NiSO ₄
	FEED RATE (KG / HR)	FEED RATE (KG / HR)	FEED RATE (KG / HR)	FEED RATE (KG / HR)	FEED RATE (KG / HR)	FEED RATE (KG / HR)
3,400	3,264	136	3,287	113	3,303	97
3,500	3,360	140	3,383	117	3,400	100
3,600	3,456	144	3,480	120	3,497	103
3,700	3,552	148	3,577	123	3,594	106
3,800	3,648	152	3,673	127	3,691	109
3,900	3,744	156	3,770	130	3,789	111
4,000	3,840	160	3,867	133	3,886	114
4,100	3,936	164	3,963	137	3,983	117
4,200	4,032	168	4,060	140	4,080	120
4,300	4,128	172	4,157	143	4,177	123
4,400	4,224	176	4,253	147	4,274	126
4,500	4,320	180	4,350	150	4,371	129
4,600	4,416	184	4,447	153	4,468	132

Figure 14, Photograph of rate parameters for a 25:1 feed ratio for a total feed rate between 3,400 and 4,600 kg per hour.

Equation 1, Calculation to verify Sulphur content for total mass (Francis, 2024).

T_{rate} = Total hourly feed rate

NS_{rate} = Hourly feedrate of NiSO₄.6H₂O

S_E = Estimated Sulphur content of Total

S_p = Decimal percentage of Sulphur in 1
mass unit of NiSO₄.6H₂O

$$\frac{\left(\frac{S_p}{100} \times NS_{rate}\right)}{T_{rate}} \times 100 = S_E$$

$$\frac{\left(\frac{12.198}{100} \times 80\right)}{2000} \times 100 = 0.488\%$$

Equation 2, Analytical method to verify Nickel content in Nickel Sulphate Hexahydrate

NS_m = Total NiSO₄.6H₂O mass

NA_m = Total Nickel atomic mass

N_p = Calculated Nickel percentage

$$\frac{NA_m}{NS_m} \times 100 = N_p$$

$$\frac{58.6934}{262.87} \times 100 = 22.327\%$$

Equation 3, Calculation to consider the affect to sulphur content in a consignment that contains impurities

$$N_v = 90 \% \text{ Volume of } \text{NiSO}_4 \cdot 6\text{H}_2\text{O} \text{ Consignment}$$

$$N_d = \text{Density of } \text{NiSO}_4 \cdot 6\text{H}_2\text{O}$$

$$S_m = \text{Sulphur content mass of } \text{NiSO}_4 \cdot 6\text{H}_2\text{O}$$

$$S_p = \text{Decimal percentage of Sulphur in 1 mass unit of } \text{NiSO}_4 \cdot 6\text{H}_2\text{O}$$

$$\left(\frac{N_v \times N_d}{1000} \right) \times S_p = S_m$$
$$\left(\frac{900000 \times 3.68}{1000} \right) \times 0.12198 = 404\text{Kg}$$

5. Experimental Findings

Results of our experiment are shown in Table 2, when we compare the percent of Sulphur obtained by experiment to the analytical method we have used to estimate, we see that actual Sulphur content for each sample is higher than we expected for both manufacturers. We also see variation about the mean value for the four samples which, has a range between -0.09% to +0.1%.

Furthermore, we see that there are variations between the result of each pH test with both deliveries received from Umicore being 33% more acidic than the deliveries received from Bluestone. We calculate the effect of an increase to; the proportion of Sulphur in one mass unit of $NiSO_4 \cdot 6H_2O$, we use a mean value of the Sulphur recorded in Table 2 (12.733%) then, use Equation 1 as the foundation to generate Equation 4 which shall provide a basis for comparison. Expectingly we find that Sulphur content as a proportion of the total material fed into the Hydrogen reduction process increases from 0.488 % to 0.509.

Table 2, Test Results (Jayne, 2024)

Batch Number	Manufacturer	Date of test	pH	% S
23-143V	Bluestone	24/04/2024	6.1	12.71163
23-399V	Bluestone	24/04/2024	6.1	12.84305
2940502620	Umicore	24/04/2024	4.1	12.6467
2940502697	Umicore	24/04/2024	4	12.73358

Equation 4, Calculation of Actual Sulphur proportion of the total material fed into the Hydrogen Reduction Process

T_{rate} = Total hourly feed rate

NS_{rate} = Hourly feedrate of $NiSO_4 \cdot 6H_2O$

S_E = Estimated Sulphur content of Total

S_p = Decimal percentage of Sulphur in 1
mass unit of $NiSO_4 \cdot 6H_2O$

$$\frac{\left(\frac{S_p}{100} \times NS_{rate}\right)}{T_{rate}} \times 100 = S_E$$

$$\frac{\left(\frac{12.733}{100} \times 80\right)}{2000} \times 100 = 0.509\%$$

6. Discussion and Conclusion

The author is content that their report has identified potential causes by means of population sampling however, they know that it would not provide substantial evidence that would endorse reasons for change to the way Vale's production team operate, for this, we need to create an adequate process sampling plan. In conjunction with this we need to establish the processes Lower Control Limit (LCL) and, the processes Upper control Limit (UCL) these ensure that our process operators can identify common causes of variation from the special causes of variation.

We review Figure 11 and see that, changing the manufacturer of the $NiSO_4 \cdot 6H_2O$ used by the process has an impact to the measurement of percentage of Nickel Oxide that was not reduced. Our results show that actual Sulphur content from the samples which were taken from consignment lots, were higher than that found by the analytical method, according to theory this should not impact the rate at which Nickel Oxide is Reduced therefore, would not have an influence on the percentage unreduced metric hence, an increase to the temperature of Hydrogen gas exiting Kiln 2 Born Heater would not be required. That said, we have great concerns regarding the location of which samples are taken to analyse for the percentage of unreduced Nickel Oxide which remains after the Hydrogen reduction process. At present these are taken after the material has left the Activation Kiln, a process of which, according to Vale Europe Ltd. (2023), little is known about the chemical reaction that takes place therefore, we can not be confident that this reaction does not influence both the percentage of unreduced Nickel Oxide metric and, the SSA metric.

Analysis of pH levels in our samples were included in the experiment as merely an opportunity for the author to enhance their knowledge of the impact that certain pH levels may have on the integrity of assets which, is a responsibility for their current role in the organisation however, when we compare and contrast Figure 11 with Table 2 we see correlation that the more acidic compound received from Umicore increases the percentage of Nickel Oxide that has not been reduced, this is based on the assumption that the compound used at the time of production was acidic – we do not have evidence that this is true. Table 3 and Table 4 show results for the amount of Nickel Oxide that was not reduced, during an equal period before and after changing from Umicore manufactured $NiSO_4 \cdot 6H_2O$ to the Bluestone manufactured compound, we see that; during the period that the process uses the compound manufactured by Umicore, the percentage of unreduced Nickel Oxide was 66% higher than that of a period when a Bluestone compound was used. To contextualize, we consider the annual waste that may be produced whilst feeding the blended TNOS and $NiSO_4 \cdot 6H_2O$ material at a rate of 4 tonnes an hour then, assign a monetary value to the accumulation of the waste which, provides an annual cost incurred by the organisation. We see from Equation 5 that if the percentage of unreduced Nickel Oxide were to be reduced to that of the level whilst using Bluestone manufactured $NiSO_4 \cdot 6H_2O$, an additional 760,228 kg of activated Nickel would have been processed by the Volatilisation Kiln and, based on current extraction rates of 90%, 684,205 kg of grade 1 Nickel would have been produce that, according to the prices at the time of writing, we could estimate (Equation 5), to have a value of £10,447,811.21 (LME, 2024).

Though we must be cautious, our concern is; we have narrowed our scope by analysing only two of the process parameters at the time of the change, we must consider all parameters that may influence however, we feel that the current sampling regime does not provide this data nonetheless, we feel that considering the potential savings which could be made, further work is necessary.

When we are confident with the sampling plan and, find consistent results which, endorse the results found by this report, there may be potential saving opportunities that may benefit the business. We reflect on Equation 4 and make comparisons to theory, we find; the Hydrogen reduction process has

more than sufficient Sulphur to enable an effective process therefore it would be prudent to adjust the feed rate of Nickel Sulphate Hexahydrate to a measure which provides the value of Sulphur obtained by the analytical method. We consider the effects of this adjustment in Equation 6 and see that there may be potential to reduce the feed rate of $NiSO_4 \cdot 6H_2O$, to 77 kg per hour which would accumulate to an annual reduction of 26,280 kg, based on the current prices this would have a monetary value of £90,140.40 which, may also be applicable to Kiln line 1 therefore, doubling the savings.

Our work is inclusive, although contrary to Laker, (2008), our data suggests that the effects Nickel Sulphate pH levels are not limited to; an increase in corrosion of pipework throughout the hydrogen circuit but, there seems to be a tangible relationship with the quantity of Nickel Oxide which had not been reduced. We believe that further work is required to first; establish a sampling programme which is consistent and, relevant to the process of which to be monitored, secondly; to test each sample and collate data for analysis.

Table 3, Average Percentage of Nickel that was unreduced during a campaign of Nickel Sulphate Hexahydrate manufactured by Umicore

Date	Sulphate type	% Unreduced Nickel (-48hrs Offset)
08/07/2023	UMICORE	3.57
09/07/2023	UMICORE	1.6
10/07/2023	UMICORE	0.42
11/07/2023	UMICORE	1.21
12/07/2023	UMICORE	5.56
15/07/2023	UMICORE	3.66
16/07/2023	UMICORE	3.34
17/07/2023	UMICORE	2.57
18/07/2023	UMICORE	1.99
19/07/2023	UMICORE	4.24
22/07/2023	UMICORE	3.35
23/07/2023	UMICORE	1.93
24/07/2023	UMICORE	5.25
25/07/2023	UMICORE	3.04
26/07/2023	UMICORE	2.61
29/07/2023	UMICORE	4.15
30/07/2023	UMICORE	9.33
31/07/2023	UMICORE	8.58
01/08/2023	UMICORE	1.94
05/08/2023	UMICORE	4.42
06/08/2023	UMICORE	1.79
07/08/2023	UMICORE	1.97
08/08/2023	UMICORE	0.68
09/08/2023	UMICORE	4.56
% Unreduced Average:		3.41

Table 4, Average Percentage of Nickel that was unreduced during a campaign of Nickel Sulphate Hexahydrate manufactured by Bluestone

Date	Sulphate type	% Unreduced Nickel (-48hrs Offset)
12/08/2023	BLUESTONE	0.56
13/08/2023	BLUESTONE	0.87
14/08/2023	BLUESTONE	1.13
15/08/2023	BLUESTONE	1.05
16/08/2023	BLUESTONE	0.85
19/08/2023	BLUESTONE	0.99
20/08/2023	BLUESTONE	1.59
21/08/2023	BLUESTONE	1.32
22/08/2023	BLUESTONE	1.34
23/08/2023	BLUESTONE	0.76
27/08/2023	BLUESTONE	0.67
28/08/2023	BLUESTONE	0.74
29/08/2023	BLUESTONE	0.47
30/08/2023	BLUESTONE	0.27
02/09/2023	BLUESTONE	1.99
03/09/2023	BLUESTONE	1.24
04/09/2023	BLUESTONE	1.78
05/09/2023	BLUESTONE	1.61
06/09/2023	BLUESTONE	0.68
09/09/2023	BLUESTONE	0.6
10/09/2023	BLUESTONE	1.68
11/09/2023	BLUESTONE	2.25
12/09/2023	BLUESTONE	2.4
13/09/2023	BLUESTONE	0.76
% Unreduced Average:		1.15

Equation 5, A calculation to find the potential value of waste created by using Umicore Nickel Sulphate Hexahydrate

T_R = TNOS hourly feed rate

T_y = TNOS feed rate

P_h = Number of daily production Hours

P_d = Number of yearly production days

UR_d = Deficit of Unreduced average

V_e = Decimal percentage of Nickel Extracted

N_p = price of grade 1 Nickel per Kilogram

C_w = Yearly cost of waste incurred

$$\left(\frac{T_R \times P_h \times P_d \times UR_d \times V_e}{100} \right) \times N_p = C_w$$

$$\left(\frac{3840 \times 24 \times 365 \times 2.26 \times 0.9}{100} \right) \times £15.27$$

$$= £10,447,811.21$$

Equation 6, Calculation to find adjusted feed rate of Nickel Sulphate Hexahydrate

T_{rate} = Total hourly feed rate

NS_{rate} = Hourly feedrate of $NiSO_4 \cdot 6H_2O$

S_E = Estimated Sulphur content of Total

S_p = Decimal percentage of Sulphur in 1

mass unit of $NiSO_4 \cdot 6H_2O$

$$\frac{\left(\frac{S_p}{100} \times NS_{rate} \right)}{T_{rate}} \times 100 = S_E$$

$$\frac{\left(\frac{12.733}{100} \times a \right)}{2000} \times 100 = 0.488\%$$

Transpose to find 'a',

$$\frac{\left(\frac{0.488}{100} \times 2000 \right)}{12.733} \times 100 = 76.651 \text{ Kg/Hr}$$

7. Further Work

Our Discussion and Conclusion section underlined that current sampling regimes for $NiSO_4 \cdot 6H_2O$ do not exist, we believe that if a sample point were to be installed on pipework located just before the point of which the blended feed material enters into the Reduction Kiln which has been illustrated as “Sample Point 1” on Figure 16, this would constitute an adequate process sampling plan which would allow daily analysis for Sulphur content.

The provision of a UCL and, an LCL which are visible on a Control Chart that is used to record Sulphur results would provide a process operator with the information they would need to adjust $NiSO_4 \cdot 6H_2O$ feed rates appropriately. Establishing the UCL and, the LCL must take into consideration, process effectiveness and cost and, must be endorsed by the Process Extraction Superintendent. We can determine the LCL to be the value of 0.4% which Foran (2014), finds to be the most effective amount of Sulphur needed for the Hydrogen reduction process, the UCL shall consider the additional cost to the budget if an unnecessary amount of Sulphur was fed into the process, we can consider the value of 0.488 which has been calculated in Equation 1 and, is the current method of estimating Sulphur content. We can see from the Control Chart illustrated in Figure 15 that; common causes of defects are those which stay within the limits and, special causes of defects are those which fall outside of the limits.

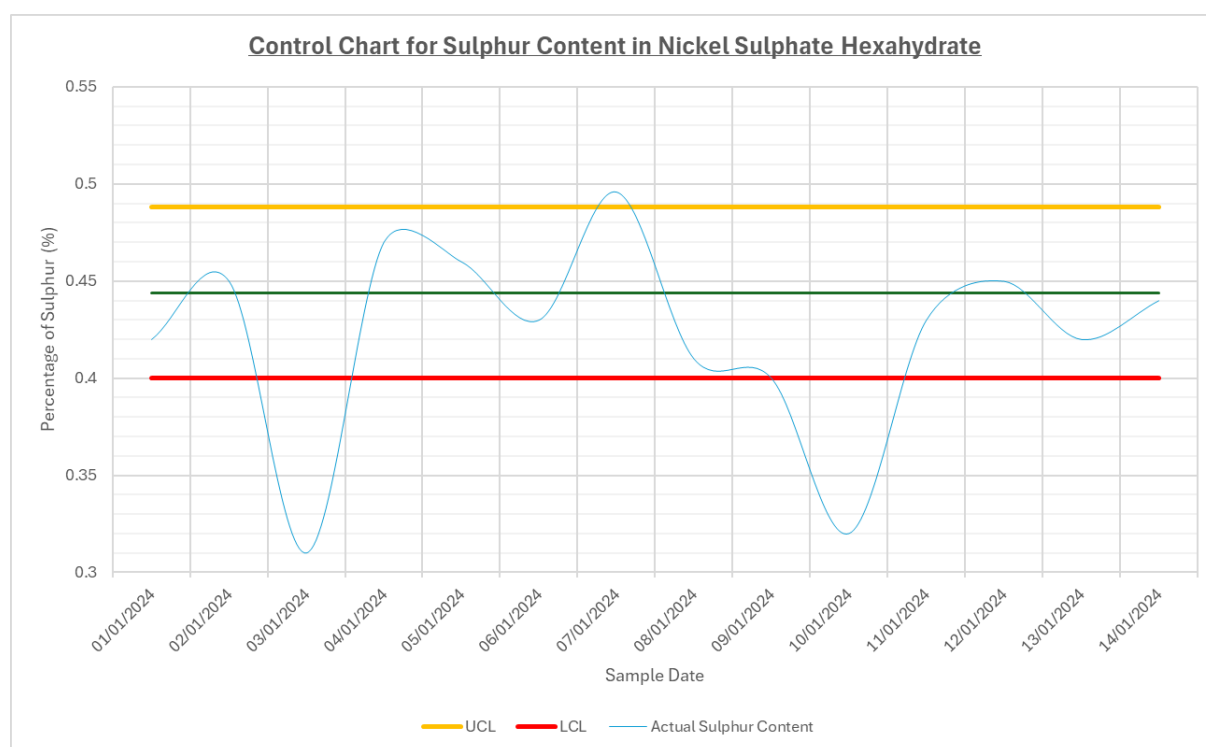


Figure 15, Control Chart for Sulphur Content in Nickel Sulphate Hexahydrate

We have found the location of which samples are taken to analyse for the percentage of Nickel Oxide that had not been reduced to be incorrect, at present, samples are taken from material which is released from the Activation Kiln a process step after the process step we wish to monitor, this contradicts Trottier & Dhodapkar (2012), advise for sampling particulate materials the right way who suggest that material should be taken at the end of every process to ensure that; relevant process variables can be adjusted in a timely fashion to rectify any defects that have been found. We believe that if a sample point was to be installed in the location illustrate as “Sample Point 2” on Figure 16, the Hydrogen reduction process could be analysed effectively with any process adjustments required to prevent a defect in quality, made earlier by the omission of the time taken for the Activation Kiln to process the material.

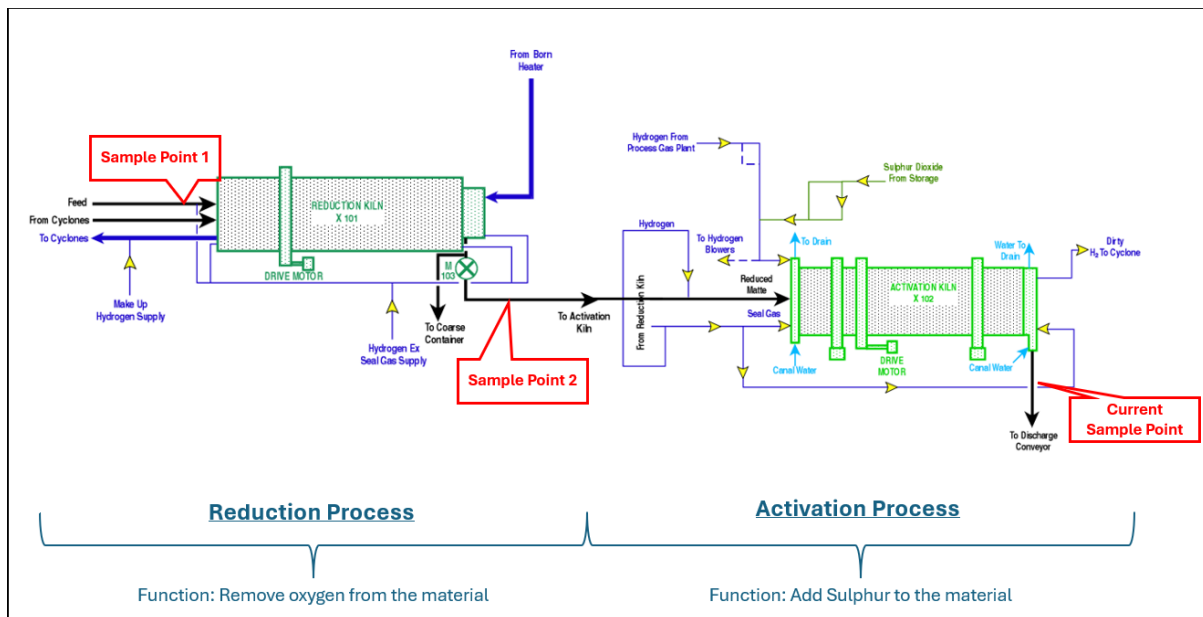


Figure 16, Identification of proposed and current sample point in the Reduction and Activation Step

We have analytically proven that by decreasing the amount of $NiSO_4 \cdot 6H_2O$ used by a relatively small amount we would be able to make substantial savings however, we have not considered the consequence of this to the process in its entirety. As we have found in our research, the production of carbonyl gas in the Volatilisation Kiln is reliant upon the amount of Sulphur adsorbed onto a Nickel molecule during its time in the Activation Kiln, if a net decrease of Sulphur addition was made by decreasing the $NiSO_4 \cdot 6H_2O$ feed rate into the Reduction Kiln, would the affect be; a reduction of activated material leaving the Activation Kiln? To answer our question, we would like to make a small incremental reduction to the Feed rate of Nickel Sulphate Hexahydrate whilst, monitoring the content of Sulphur entering the Reduction Kiln and, after allowing a period for the material to journey through the Reduction and Activation Kiln, we would like to sample to analyse the amount of Sulphur present.

Although monitoring process variables and adjusting parameters will provide an effective process it shall still remain susceptible to human factors which, for this scenario, will have the potential to create defects and waste if an operator selected a disproportionate feed ratio of $NiSO_4 \cdot 6H_2O$ for the feed rate of the TNOS. We believe that this error could be prevented if the system were to be automated by taking the 0-5 Volt control signal for the VSD's driving the discharge systems from the TNOS weigh hopper and, the $NiSO_4 \cdot 6H_2O$ which, at present are adjusted by a potentiometer, to the DSC.

This report has not found conclusive evidence which supports that; pH has an influence on the Hydrogen Reduction Process, we would like to further explore this by devising a laboratory experiment whereby, we adapt a current Technical department Standard Work Procedure to determine the amount of Nickel Oxide that has not been reduced when Nickel Sulphate Hexahydrate of various pH values are blended with TNOS and used in the Hydrogen reduction process. This experiment will use the 'Laboratory Scale Carbonylation: Reduction Step' apparatus which will mimic said process, a representative sample of 50g shall be taken from a lot of Nickel Sulphate Hexahydrate then, a quantity of 1.64g shall be added to a beaker whereby, 5cm³ of pH3 buffer solution is added, the beaker is then placed on a hot plate to dissolve. Once dissolved, 50g of Nickel Oxide is added and stirred then, will be left on a plate to dry out. A test tube shall be used to contain the sample in a void of 10cm long which shall be created by two glass wool plugs, the tube shall then be connected to a rubber tube then, placed into a cradle which is suspended in the furnace and attached to a weigh scale. The furnace shall be switched on and, heated to a temperature of 420°C where it shall remain at throughout this experiment. The weigh scale shall be zeroed, this will be used to determine the end of the reduction process, Hydrogen gas shall then be introduced via a needle plug valve where the flow shall be limited to an equivalent of 15 L.h⁻¹. The reduction process will be deemed complete once the weigh scale no longer registers a loss in weight which typically would take 15 minutes to process, the sample shall then be removed and cooled. The process shall be repeated for a pH buffer solution with pH values of 4, 5, 6, once all tests have been complete they shall be sent to the quality assurance laboratory where they will analyse for the percentage of unreduced Nickel Oxide remaining.

Bibliography

- Averill, B. A. & Eldredge, P., 2012. *Principles of General Chemistry*. 1 ed. s.l.:Creative Commons.
- Bevan, A., 2024. *Kiln Plant Team Leader* [Interview] (24 February 2024).
- Brown, T. L., 2015. 4.4: Oxidation-Reduction Reactions. In: *Chemistry: The Central Science*. 1 ed. s.l.:Boston: Pearson.
- Cameo Chemicals, 2024. *NICKEL SULFATE HEXAHYDRATE*. [Online]
Available at: <https://cameochemicals.noaa.gov/chemical/20735>
[Accessed 24 April 2024].
- Chemical Aid, 2024. *NiSO₄*6H₂O (Nickel Sulfate Hexahydrate) Molar Mass*. [Online]
Available at:
<https://www.chemicalaid.com/tools/molarmass.php?formula=NiSO4%2A6H2O&hl=en#:~:text=To%20find%20the%20mole%20fraction%20and%20percentage%20of,%3D%200.223298%20%C3%97%20100%20%3D%2022.3298%25%20S%20%28Sulphur%2FSulfur%29>
[Accessed 20 April 2024].
- CR Plus Ltd, 2013. *Assessment of Energy Saving Opportunities for Vale Clydach Nickel Refinery Clydach, South Wales*., Rugeley, Staffordshire: CR Plus.
- Croft, D., 2024. *Learn Lean6Sigma*. [Online]
Available at: <https://www.learnleansigma.com/guides/impact-effort-matrix/>
[Accessed 16 March 2024].
- Dalling, R., 2019. *The big refinery in the middle of a Welsh village that has kept a community alive for a century*. [Online]
Available at: <https://www.walesonline.co.uk/news/wales-news/big-refinery-middle-welsh-village-16130138>
[Accessed 10 May 2024].
- D'Angeli, J., 2024. *Process Extraction co-ordinator* [Interview] (10 January 2024).
- Ebenezer, D., 2024. *Kiln Plant Team Leader* [Interview] (17 February 2024).
- Foran, G., 2014. *Nickel Oxide Reduction and the Effect of Metal Sulfates*, Guelph, Ontario: University of Guelph.
- Foxboro DSC, 2024. *Kiln 1 and Kiln 2 I/A Graphics* , Swansea, Wales: Vale Europe Ltd..
- Francis, M., 2024. *Vale Europe Ltd. Technical Quality Supervisor* [Interview] (5 March 2024).
- Fussell, R., 2023. *Process Gass Plant Superintendent* [Interview] (14 December 2023).
- GlobalPetrolPrices.com, 2023. *Natural Gas Prices*. [Online]
Available at: https://www.globalpetrolprices.com/natural_gas_prices/
[Accessed 09 February 2024].
- Greig, P. A., 1972. *The effect of nickel extractions of the time and temperature of reduction of F.M.W, with and without nickel sulphate*, Swansea, Wales: International Nickel Limited .
- Jackman, J., 2023. *EcoExperts*. [Online]
Available at: <https://www.theecoexperts.co.uk/blog/reasons-for-uk-gas-price-increase>
[Accessed 04 February 2024].
- Jayne, A., 2024. *Quality assurance Laboratory Supervisor* [Interview] (02 May 2024).
- Jolly, W. L., 2024. *Reactivity of Hydrogen*. [Online]
Available at: <https://www.britannica.com/science/hydrogen>
[Accessed 25 February 2024].

- Jones, A., 2024. *Integrated Business Planner (IBP)* [Interview] (19 January 2024).
- Jones, G., 2023. *Mechanical Superintendent* [Interview] (1 November 2023).
- Kathiravan, P. et al., 2016. Growth and characterization of α -nickel sulphate hexahydrate single crystal. *Karbala International Journal of Modern Science*, 2(4), pp. 226-238.
- Laker, R., 2008. *Reduction/Carbonylation testwork of FMW with Nickel Sulphate from Different Suppliers*, Swansea: INCO Europe Limited.
- Lascelles, K., 1986. *Sulphur and the carbonylations of Nickel*, Swansea: Vale Europe Ltd.
- Lascelles, K., 2004. *Alternatives to the present nickel sulphate in the kiln plant*, Swansea: Vale Europe Ltd.
- Law & Your Environment, 2024. *Environmental, social and governance*. [Online]
Available at: <https://environmentlaw.org.uk/LYE/LYE/ESG/ESG-home.aspx>
[Accessed 3 May 2024].
- Lee, D. S. & min, D. J., 2019. A kinetics of Hydrogen Reduction of Nickel Oxide at Moderate Temperature. *Metals and Materials International*, Volume 25, pp. 982-990.
- Lewis, P., 2023. *Process Extraction Plant team Leader* [Interview] (16 November 2023).
- LME, 2024. *LME Nickel*. [Online]
Available at: <https://www.lme.com/Metals/Non-ferrous/LME-Nickel#Summary>
[Accessed 02 February 2024].
- Lu, X. et al., 2021. Metal-Free Phosphated Mesoporous SiO₂ as Catalyst for the Low-Temperature Conversion of SO₂ to H₂S in Hydrogen.. *Nanomaterials*, 11(9), p. 2440.
- Moore, M., 2015. *Effect of the composition, Nickel Sulfate Addition, and Reduction Temperature on the Morphology of Nickel Oxide Through Reduction*, Guelph, Ontario, Canada: University of Guelph.
- Moore, M., 2015. *Effect of the Composition, Nickel Sulfate Addition, and Reduction Temperature on the Morphology of Nickel Oxide through Reduction*, Guelph, Ontario: University of Guelph.
- Nasrazadani, S. & Hassani, S., 2016. *Modern analytical techniques in failure analysis of aerospace, chemical, and oil and gas industries*. 1 ed. s.l.:Butterworth-Heinemann.
- National Center for Biotechnology Information, 2024. *Nickel*. [Online]
Available at: <https://pubchem.ncbi.nlm.nih.gov/compound/nickel>
[Accessed 5 May 2024].
- National Center for Biotechnology Information, 2024. *Nickel sulfate hexahydrate*. [Online]
Available at: <https://pubchem.ncbi.nlm.nih.gov/compound/Nickel-sulfate-hexahydrate#section=Use-Classification>
[Accessed 5 May 2024].
- National Library of Medicine, 2024. *Nickel sulfate hexahydrate*. [Online]
Available at: <https://pubchem.ncbi.nlm.nih.gov/compound/Nickel-sulfate-hexahydrate#section=Information-Sources>
[Accessed 22 March 2024].
- Nutburn, M., 2024. *ISO: Who, What, Why and How?*. [Online]
Available at: <https://www.british-assessment.co.uk/insights/who-are-iso-and-why-do-they-exist/#:~:text=Businesses%20use%20ISO%20International%20Standards%20as%20strategic%20tools,businesses%20deliver%20the%20same%20consistency%20to%20their%20clients.>
[Accessed 03 May 2024].

- Oswa, T., Hamano, Y., Saga, S. & Takayasu, O., 2008. Hydrogen-deuterium exchange of methane on nickel surface prepared by the reduction of nickel oxide. *Journal of Molecular Catalysis A: Chemical*, 298(2009), pp. 111-114.
- Pigott, P. & Price, B., 2024. *Tata Steel is to push ahead with plans to close both its blast furnaces in Port Talbot with the expected loss of 3,000 jobs UK-wide, the BBC has been told.*, Swansea: BBC.
- Plascenia, G. & Utigard, T., 2009. The Reduction of Tokyo and Sinter 75 nickel oxides with Hydrogen. *Chemical Engineering Science*, Volume 64, pp. 3879-3888.
- Poland, J., 2023. *Process Extraction Superintendent* [Interview] (20 November 2023).
- Roselina, N. N. et al., 2013. Effect of pH on Formation of Nickel Nanostructures through Chemical Reduction Method. *Procedia Engineering*, Volume 68, pp. 43-48.
- Science Notes, 2024. *Reducing Agent (Reductant) Definition and Examples*. [Online]
Available at: <https://sciencenotes.org/reducing-agent-reductant-definition-and-examples/#:~:text=Examples%20of%20reducing%20agents%20include%20hydrogen%20gas%2C%20alkali,occur%20together%2C%20the%20reaction%20is%20a%20redox%20reaction.>
[Accessed 9 May 2024].
- Sterling, C., 2023. *Technologist* [Interview] (28 November 2023).
- Tomaszewicz, E. & Kotfica, M., 2004. MECHANISM AND KINETICS OF THERMAL DECOMPOSITION OF NICKEL(II) SULFATE(VI) HEXAHYDRATE. *Journal of Thermal Analysis & Calorimetry*, Volume 77, pp. 25-31.
- Trottier, R. & Dhodapkar, S., 2012. *Sampling Particulate Materials the Right Way*. [Online]
Available at: <https://www.chemengonline.com/sampling-particulate-materials-the-right-way/>
[Accessed 12 May 2024].
- Vale Europe Ltd., 2023. *OV200 Overview Process Training Manual*, Swansea, Wales: Vale Europe Ltd..
- Vale Europe Ltd., 2023. *PSO202 Reducing & Activating Process Manual*, Swansea: Vale Europe Ltd..
- Vale Europe Ltd., 2024. *Bonham*, Swansea, Wales: Vale Europe Ltd..
- Vale Europe Ltd., 2024. *PS203 Volatilisation Process Manual*, Swansea, Wales: 2023.
- Vale Europe Ltd., 2024. *Transforming the future – Vale in Wales, U.K.*. [Online]
Available at: <https://www.bing.com/search?q=the+mond+nickel+refinery&qsn&form=QBRE&sp=-1&ghc=1&lq=0&pq=the+mond+nickel+refinery&sc=10-24&sk=&cvid=969547FFA3744B3499789D8870E0F3B2&ghsh=0&ghacc=0&ghpl=>
[Accessed 11 May 2024].
- Zhang, M., Fu, Z. & Yu, Y., 2019. Adsorption and decomposition of H₂S on the Ni(1 1 1) and Ni(2 1 1). *Applied Surface Science*, Volume 473, pp. 657-667.

Appendix A

This section includes a report detailing the findings of an investigation into energy inefficiencies of Kiln Line 1 and, Kiln Line 2 Born Heater.



RESTRICTED – COMMERCIAL

**Carbon Management Energy Efficiency
Covering Report**

**Assessment Of Energy Saving Opportunities For
Vale Clydach Nickel Refinery
Clydach, South Wales**

Prepared for	Alexander McDowall Energy Superintendent Vale Europe Limited Clydach Refinery Clydach Swansea SA6 5QR	Prepared by	Sarah Rees, Jamie Jones, Richard Pyatt & Tony Parton CR Plus Ltd PO Box 5094 Rugeley Staffordshire WS15 9BN
Tel	01792 842501	Tel	07970 957858
Email	Alexander.Mcdowall@vale.com	Email	tparton@crplus.co.uk
Account manager	David Moore	Tel	01443 845947 07714 684338
		Email	David.Moore@carbontrust.co.uk
Product ID	P-0144752		
PO Reference	PO038179		
Country	Wales		
Date	08 th February 2013		
Version	1.0 Final		



Carbon Management Energy Efficiency Report for Vale Europe Limited, Clydach Nickel Refinery
08th February 2013 by CR Plus Ltd

CONTENTS

EXECUTIVE SUMMARY	3
1. INTRODUCTION	5
2. BENCHMARKING RESULTS	7
3. SAVINGS POTENTIAL	13
4. AFTER THE REPORT	15
APPENDIX 1: INITIAL EMAIL QUOTE – BORN	16



EXECUTIVE SUMMARY

This report presents the results of a Carbon Management Energy Efficiency (CMEE) survey of Vale Europe Limited, Clydach Nickel Refinery carried out by CR Plus Ltd. The survey and report are provided by the Carbon Trust. The Carbon Trust receives public funding from the Scottish Government and the Welsh Government.

The current agreed scope of work is to undertake the objectives set out which includes a review the site's Born heaters and other heaters based on benchmark analysis and to research other plant heating options with the compilation of the outline business case for Born heaters.

Two Born heaters are located on the roof of the Kiln Plant at Vale Clydach (one per production line); their purpose is to pre-heat hydrogen for delivery to the Reduction Kiln. Within the Reduction Kiln this pre-heated hydrogen is used as a reducing gas, removing oxygen from the nickel oxide feedstock by reacting to form water vapour and therefore leaving a nickel reduced 'matte', which is fed into the Activation Kiln for further processing. The water vapour exits in the kiln suspended in hot gas which is processed through a series of cyclones and scrubbers before being cooled. From this clean hydrogen is then fed back into the Born Heater for pre-heating to continue the recirculation loop.

Heat energy (into the Born Heaters) is created from burning natural gas as the fuel; this energy is transferred to three main outputs:

1. Hydrogen Heating
2. Surface Heat Loss/ Radiation
3. Stack Waste Heat

Minimising the latter two outputs within any new schemes is a key consideration to any efficient plant. The Born Heaters use **33.3GWh** of energy per year at a cost of **£749k**.

Quick Win

Born Heater 2 uses substantially more gas than Born Heater 1 and although the additional hydrogen flowing through No.2 heater accounts for 7% additional gas the rest is due to higher stack loss and higher structural heat loss. It was noted that gaps existed between the convection heat exchanger and the supporting structure as the insulation was removed; this both cooled the hydrogen and prevented heat transfer from the combustion gases prior to expulsion to atmosphere (high exhaust temperature with additional oxygen). In addition the hot hydrogen pipework emits heat. Sealing this area will optimise energy efficiency in relation to both aspects and save circa £36k per year.

Access can be obtained but at that height and obvious health and safety implications the cost of repair will probably amount to £20K (still less than a 1 year payback).

Long Term Born Heater Replacement Benefits

The only other real reduction in energy is to purchase two new Born heaters that will incorporate the highest insulation standards and extract as much heat from the exhaust gases by the convection heat exchanger or a combustion air heat exchanger whilst not dipping below the gas dew point. Preliminary calculations and initial costings suggest savings would amount to £195k per year for a £1.9m Capex i.e. a 10 year payback. This may support the justification of new Born heaters when required on maintenance or health and safety grounds.

Overall Package

When implemented, the Born heater system specified in this report is estimated to achieve a **31%** saving. This represents circa **£231K** per year on an on-going basis. The cost of implementing the package of measures is estimated to be circa **£1.9m** giving a simple payback period of 10 years.

Estimated Carbon Savings. Overall saving will be **1,891 tCO₂/per year**.

IMPORTANT NOTICE

All costs and savings stated in this report are based on the data available at the time of the visit.
For further liability information, see the Important Notice section.

1. INTRODUCTION

IMPORTANT NOTICE: Whilst reasonable steps have been taken to ensure that the information contained within this Carbon Management Energy Efficiency (CMEE) Report is correct, you should be aware that the information contained within it may be incomplete, inaccurate or may have become out of date. Accordingly, CR Plus Ltd, the Carbon Trust, its agents, contractors and sub-contractors and the Government make no warranties or representations of any kind as to the content of this < Carbon Management Energy Efficiency (CMEE) Report Carbon Management Energy Efficiency (CMEE) Report or its accuracy and, to the maximum extent permitted by law, accept no liability whatsoever for the same including without limit, for direct, indirect or consequential loss, business interruption, loss of profits, production, contracts, goodwill or anticipated savings. Any person making use of this Carbon Management Energy Efficiency (CMEE) Report does so at their own risk. © Queen's Printer and Controller of HMSO. Any trademarks, service marks or logos used in this publication are the property of the Carbon Trust, and copyright is licensed to the Carbon Trust. Nothing in this publication shall be construed as granting any licence or right to use or reproduce any of the trademarks, service marks, logos, copyright or any proprietary information in any way without the Carbon Trust prior written permission. The Carbon Trust enforces infringements of its intellectual property rights to the full extent permitted by law.

The Carbon Trust is a company limited by guarantee and registered in England and Wales under Company Number 04190230 with its Registered Office at: 4th Floor, Dorset House, 27-45 Stamford Street, London SE1 9NT.

1.1 General

This consultation was carried out during the months of October to December by Tony Parton (TP) (lead consultant) with Richard Pyatt (RP), Jamie Jones (JJ) and Sarah Rees (SJR) of CR Plus Ltd. Our main site contacts were Alexander McDowall, Energy Superintendent and Fiona Buttrey, Technical Manager.

1.2 Organisation Background

Vale is a long established site at Clydach operating a complex process with very significant hazard potential. It has recently become part of the Brazil based Vale Corporation along with its Canadian parent company. Whilst energy efficiency has not been a major priority in the past, this is now being embraced throughout the Vale organisation and this survey is part of that initiative.

The Vale Clydach refinery consumes 286.6 million kWh of energy in a typical 12 month period costing circa £7.6 million at agreed tariff rates. This is in a factory area of 215,000m² producing nickel products (pellet or powder) in bulk.

1.3 Current Hydrogen Heating System

Two Born Heaters are located on the roof of the Kiln Plant at Vale Clydach, one per production line; their purpose is to pre-heat hydrogen for delivery to the Reduction Kiln. Within the Reduction Kiln this pre-heated hydrogen is used as a reducing gas, removing oxygen from the nickel oxide feedstock by reacting to form water vapour and therefore leaving a nickel reduced 'matte', which is fed into the Activation Kiln for further processing. The water vapour exits in the kiln suspended in hot gas which is processed through a series of cyclones and scrubbers before being cooled, this clean hydrogen is then fed back into the Born Heater for pre-heating and therefore recirculated again to the Reduction Kiln.

Heat energy into the Born Heaters is created from burning natural gas as the fuel; this energy is transferred to three main outputs:

1. Hydrogen Heating
2. Surface Heat Loss/ Radiation
3. Stack Waste Heat

Born Heater Arrangement:

Two heating chambers - a Convection Bank and a Radiant Heating Zone.

The convection bank is the first heating chamber the hydrogen enters, here the flue gas passes around four parallel (hydrogen carrying) 100mm diameter steel tubes, in horizontal formation, which meander vertically to form seven rows before exiting externally.

These four hydrogen pipes, on exiting the convection bank, turn and drop down on the outside of each heater into the top of a Radiant Heating Zone, here the four steel pipes meander vertically in a circular direction within this second and final heating chamber, where after the hydrogen is transported to the Reduction Kiln.

A sketch of this can be seen in the schematic on Page 8.

1.4 Energy Consumption Overview

The utility table below summarises the natural gas fuel use in two Born heaters and their relative importance.

Table 1: Energy and Related Costs.

Utility	Energy Consumption	Cost	
	kWh/year	£/year	%
Gas - Born Heater 1	14,267,761kWh	£321,025	43%
Gas - Born Heater 2	19,032,426kWh	£428,230	57%
Total Energy	33,300,187kWh	£749,255	100%

Cost figures are based on the average unit gas cost as 2.25p/kWh excluding VAT and standing charges. These values are used in any energy cost calculations. The gas and electricity costs above include the Climate Change Levy (CCL). For the avoidance of doubt, all energy terms are in delivered energy.

1.5 Objectives

The outline project objectives were to:

- Undertake the kick-off meeting
- Benchmark current system
- Risk Assessment/method statement to conduct measurement and the conducting and analysis of measurement to confirm energy balance
- Review with Clydach nickel refinery steam projects team and formulate an action plan and refine as works are in progress with further meetings to discuss and review (other sub-reports)
- Review Born heaters based on benchmark and research other plant heating options (this sub-report)
- Obtain budget supply costs and estimate additional costs
- Construct reports
- Carbon Trust DCS preparation

2.BENCHMARKING RESULTS

2.1 Structural Heat Loss Calculations

Born heater surface temperatures were measured using a Calex infra-red laser; readings were taken at regular intervals across surface sections and repeated for each heater. Using these temperature readings, surface heat losses could be calculated.

Due to the light coloured nature of the 'kiln line 1' heater, readings were significantly lower than the 'kiln line 2' heater thus considered inaccurate, therefore only readings from the 'kiln line 2' heater were used in the calculations.

BORN - Surface Heat Loss Calcs:

BORN Heaters	Area m ²	Temp °C	Heat Loss kW/m ²	Loss kW
Stack (f)	4.59	133	1.81	8.3
	4.59	170	2.69	12.4
	4.59	180	2.95	13.6
	4.59	231	4.41	20.2
Frustum (e)	3.95	130	1.75	6.9
Convection Bank (d)	7.29	151	2.21	16.1
	7.29	168	2.64	19.3
Frustum (c)	5.20	115	1.44	7.5
Main Heating Chamber (b)	6.56	102	1.18	7.8
	6.56	88	0.93	6.1
	6.56	90	0.96	6.3
	6.56	97	1.09	7.2
	6.56	93	1.03	6.7
Total - One Heater				138.4 kW
Total (x2)				276.7 kW

The above table is extracted from following imbedded file:



BORN Heater -
Surface Heat Loss.xls

*Note: It must however be noted that the calculation does not take into account the varying weather conditions these heaters are exposed to, which when poor (i.e. windy, wet and cold) dramatically multiplies the energy lost.

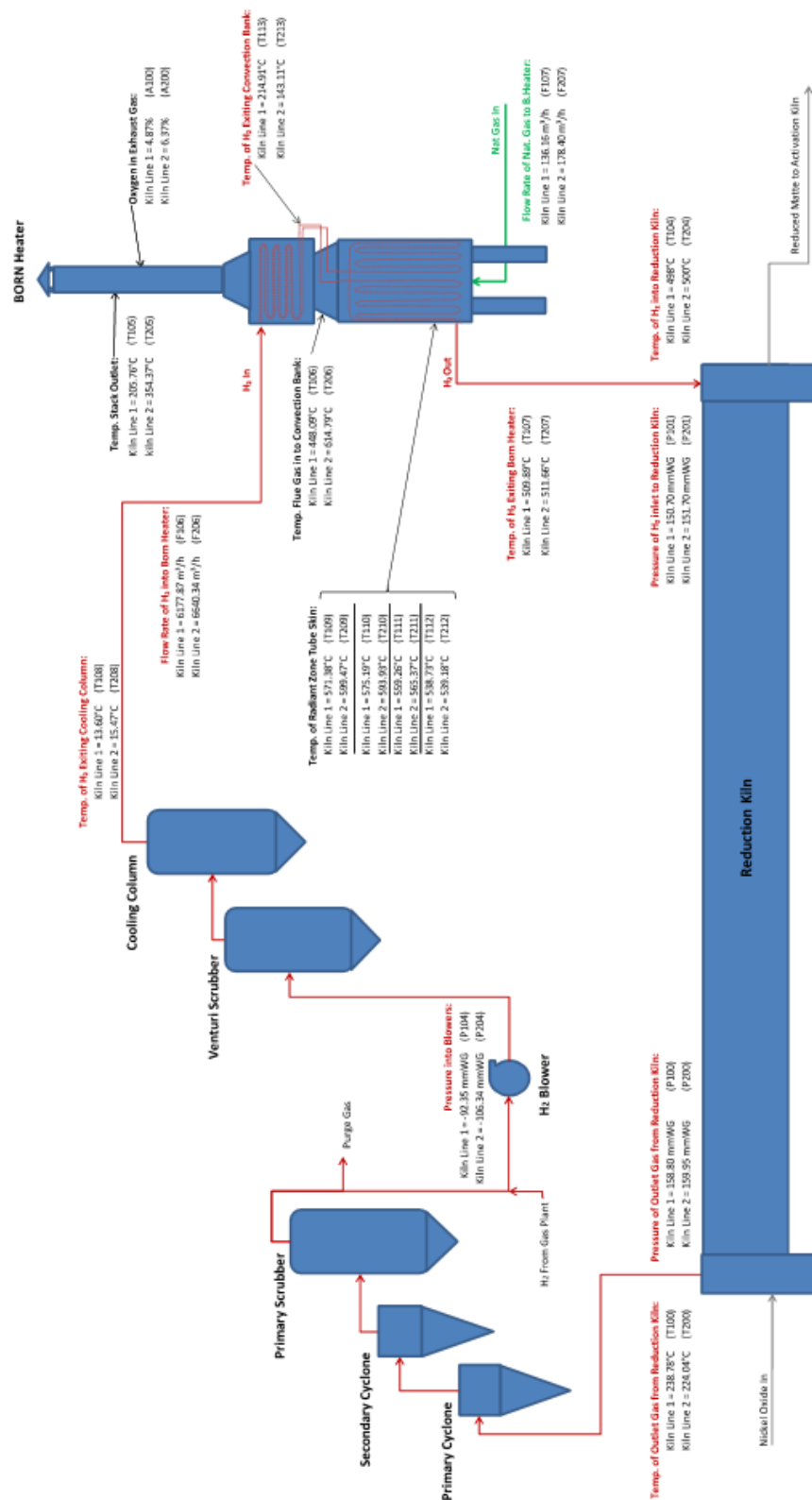
2.2 Summary of SCADA Data

SCADA data, consisting of gas temperature and flow rates to-from the Born heaters, were recorded from the Kiln Plant control room on three separate occasions:

1. 4th October,
2. 12th October and the
3. 1st November 2012.

Along with 'BONHAM' weekly data for the reduction kiln, the information obtained was used in calculating the energy consumed by the Born heaters.

Average data for both BORN heater systems are shown in the schematic below:



Using exhaust gas oxygen content and temperature, stack losses were calculated as shown below:

Stack Losses:

Kiln Line 1:	
O ₂ =	4.87 %
∴ Excess Air =	27 %
Exhaust Temp =	205.76 °C
Stack Losses =	10.4 %

Δ = 9.3 %

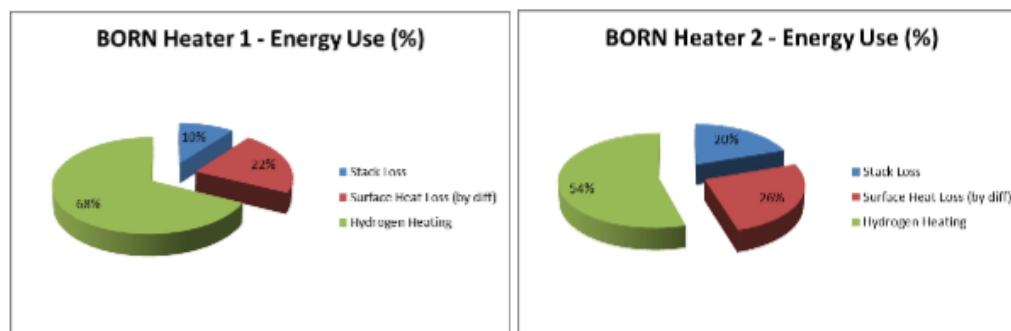
Kiln Line 2:	
O ₂ =	6.37 %
∴ Excess Air =	39 %
Exhaust Temp =	354.37 °C
Stack Losses =	19.7 %

Data for the above losses can be found in the following file:

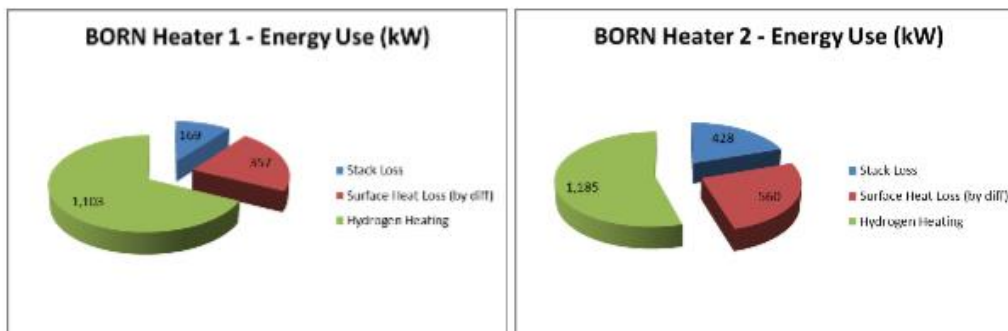


2.3 Heat Balance

When constructing the heat balance one can calculate various aspects and then assume that one element is the remainder. In this case the hydrogen flow/temperatures and the exhaust gas oxygen/temperatures are known. This leaves the structural loss, at which an attempt at an internal heat loss was attempted (section 2.1) to establish if the difference was sensible.



By percentage, heater 2 appears to be transferring less energy to hydrogen, as shown below this is not the case but instead a huge excess amount is radiated through the surface and up the stack.



The pie charts illustrated above are extracted from the following file:



Please note the effects of wind can increase surface heat loss many fold, for example at 100°C surface temperature and a 5m/s wind speed the increase in heat loss is circa 3.2. Thus the Born Heater loss in this case would increase from 138kW to 443kW. This figure is almost halfway between the kilns 1 & 2 surface heat loss estimate by difference. Also kiln 2 would lose more heat than kiln 1 as its surface is not entirely painted. Thus the surface heat loss values derived by difference are not unreasonable.



2.4 Observations and Comments

Born Heater Observations:

Both heaters are providing an overall hydrogen temperature increase of 496°C, with only a 2°C temperature difference between each line consistently, yet Born heater 2 is using more energy to do so. From the results found, it is observed that Born heater 2 is consuming 42m³/h more natural gas, at an extra cost to Vale Clydach of approximately **£95k** per year.

The Convection bank of heater 1 increases the hydrogen temperature by 201°C whereas it only adds 128°C in heater 2. Hydrogen flows at a higher rate around the No.2 reduction kiln circuit, therefore expecting more heat to be extracted; however there is missing lagging/insulation (*see photo to the right*) on this second heater and a clear gap around each tube emerging from the convection bank, part explaining the lower temperature along with the possibility of air ingress around the tubes, reducing heat exchange capability.

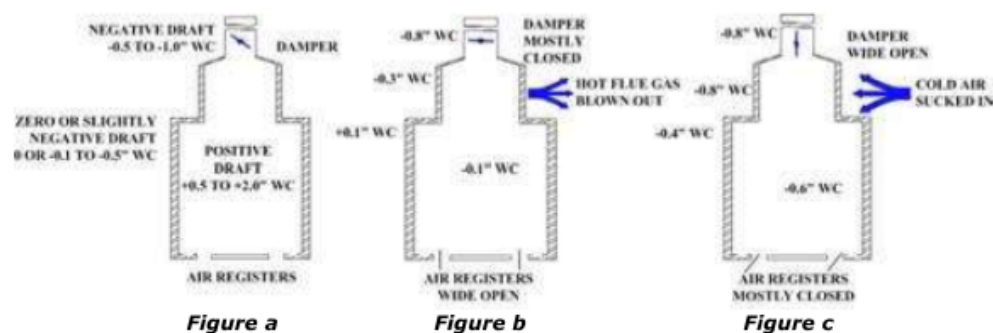


Moving next onto the Radiant Heating Zone, there are four temperature sensors coinciding with each of the four hydrogen tubes; these sensors measure each tube skin temperature, near the exit point of this heating zone (it must be noted, the label ordering of the sensors are not uniform). The temperatures observed reflect tube proximity to each of the three burners on the passage through the radiant heating zone. Data obtained shows heater No.2 is producing hydrogen of a higher

temperature; with more natural gas fed in to this burner and thus higher flue gas temperature this would be expected.

Natural gas drawn from the site ring main is fed into the natural draft gas burners; air is drawn in through a short intake duct where a damper controls the air flow. The dampers in heater No.1 burner air intake are approximately 90%, 90% and 75% open, whereas in heater No.2 are approximately 45%, 35% and 85% open, yet the No.2 heater uses more natural gas and has higher stack losses – a reflection of the exposed heater location, poor draft control (burner air intake and stack damper positioning) and also the missing lagging into the convection bank.

The higher stack oxygen percentage alone can be explained by the missing lagging allowing air ingress to the convection bank, backed by observations that the burner air intake dampers are fairly closed and stack damper is generously open* an excess negative pressure would be created in the heater, thus drawing air in, as exemplified in Figure c below:



The above Figures (a to c) demonstrate correct and incorrect heater draft, with Figures b and c reflecting excessive draft by positive and negative pressure respectively, which over time will cause damage and reduce heater efficiency.

Higher stack temperature losses in heater No.2 (almost 150°C more than No.1) are caused by the increased fuel consumption, due to the surplus negative draft increasing flow rates.

Circuit Observations:

Both hydrogen lines suffer approximately 10°C temperature losses on transition from Born heater to the reduction kiln, although both appear to be sufficiently insulated and temperatures into the kilns therefore remain approximately the same.

However the reducing gas temperature leaving each kiln differs (by 14°C), this is due not only to the higher hydrogen flow rate in the No.2 circuit but to the internal lay out of each kiln. Only 8 lifters are present in the first compartment of kiln No.2 compared to 12 in the same of kiln No.1, and this is why kiln No.2 requires a higher hydrogen flow rate - to achieve the same desired reduction as No.1. The remaining kiln chambers 2 and 3 both have 6 and 4 lifters respectively. The reason for lifter variation is unknown as this is how they were installed, but kiln No.2 was fitted at a slightly later date.

Kiln No.2 produces a higher surface area product (5.29m²/g compared to 5.24m²/g) due to more efficient oxygen extraction, this is a good measure of performance and therefore kiln No.2 is seen as the better kiln, although the cost of achieving this is high due to Born heater No.2's inefficiency.

From the 'BONHAM' data, the feed rate of nickel oxide into the kilns differs by 72kg/h (No.1 being 1.76% faster) but No.2 has a slightly higher percentage of unreduced matte leaving the reduction kiln (No.1=1.27%, No.2=1.54%)

Conclusions:

- Damper arrangements on heater No.2 are probable cause of excess negative pressure in heater (stack damper angle would need to be verified – assuming reflection of wheel bar shown right)
- Negative pressure within this heater is sucking excess air into convection bank where lagging is missing around hydrogen pipes.
- Cool air seeping into convection bank is causing lower heat exchange and higher stack oxygen percentage.
- Excess negative draft would cause more fuel consumption (natural draft gas burners)
- Higher flue gas temperature increases stack and surface heat losses
- Exposed location of these heaters (particularly No.2) escalates surface heat losses further, dramatically in wet and windy conditions



** Assuming stack damper external wheel bar is indicative of internal damper angle (see photo in 'Conclusions' Page.10)*

3. SAVINGS POTENTIAL

3.1 Quick Win – Heater 2: Convection Heater Sealing

Savings will result in two areas:

- a) The air ingress at this point is preventing the transfer of heat from the hot combustion gases to the hydrogen, thus the combustion gases are leaving this section of the convection heater hotter than unit 1 even though there is cooling air. The 70°C lack of hydrogen preheat equates to circa 14% of the heat required by the hydrogen. In reality there is no reason why Born Heater No. 2 flue gas loss should not reduce to the same percentage as No.1 heater. This will save circa 140kW of energy.
- b) Insulating the exposed sections will save circa 20kW in an internal area but as previously explained, the external wind conditions/rain will multiply this by at least a factor of 3. This will produce savings of circa 60kW on Born Heater 2.

Thus in total the 200kW saving is possible – 1,600,000 kWh or £36K.

At this moment the cost of insulation and sealing the gaps will not be significant however the cost of safely carrying out the exercise in terms of planning, scaffolding and installation will provide the bulk of the cost. This is difficult to quantify the required CapEx without calling in the relevant contractors that can accurately cost the required work in a safe manner. A CR Plus pure estimate would amount to £20K cost, however this still would provide a payback of circa 0.6 years.

3.2 Replacement Born Heater

At this stage budget quotes are still to be returned and will be passed to Vale upon receipt, but an initial email quote (Appendix 1) was used to estimate a cost of one heater below.

Items	CapEx
Removal of Existing Plant	£80,000
Project Management & Design	£90,000
CDM & HAZID plus HAZOP	£35,000
Cranes	£40,000
Born Heater (incl. Delivery)	£410,000
Installation	£100,000
Burner & WHR	£150,000
Contingency	£90,000
10%	
Total	£995,000

Thus scenario would expect the cost of two Born heaters to amount to £1.8-£2.0M.

Based on the heat balance data and specifying the following:

- Highest standard of insulation
- Improved convection box or waste heat recovery to the combustion air
- Better pressure control
- Improved fuel/air ratio

Further savings (post quick wins) could amount to 8,700,000 kWh per year or £195K between both Born heaters; i.e. an Energy Payback of circa 10 years.

4.AFTER THE REPORT

Providing Feedback

Following receipt of the final report we may ask for feedback on your experiences of working with the Carbon Trust and our consultant. The Carbon Trust values all feedback obtained.

APPENDIX 1: INITIAL EMAIL QUOTATION – BORN

"Sarah:

Please find below our budget estimate for supply of Replacement Hydrogen Heater per Born Job #1719.

HEATER: US \$510,000.00 ± 20%, per Heater, Ex-Works, Tulsa, OK, USA.

Scope of Supply for above price:

Heater Radiant Section with Coil & Refractory Installed Heater Convection Section with Coil & Refractory Installed

Heater Stack with transition section

Natural Draft Gas Burners

BURNER CONTROLS: US \$280,000.00 ± 20%, per Heater, Ex-Works, Tulsa, OK, USA.

Scope of Supply for above price:

Burner Management System w/ PLC Control Panel Fuel Gas Skid w/ Burner and pilot valve train

Delivery of the Heater will be 32 - 36 Weeks, Ex-Works. If an expedited delivery is required, please contact us.

We trust we have complied with all your requirements. If however any further details are required do not hesitate to contact us.

We appreciate your interest in Born Inc. and look forward to the possibility of working with you on this project.

Thank you.

*Best Regards,
Vijay K Rachakonda
Process Engineer
Born Inc.
Tulsa, OK 74107
www.Borninc.com*

Ph: 918.582.2186 ext: 214

Fax: 918.582.5163

Cell: 918.740.8350

CONFIDENTIALITY NOTICE:

The information contained in this email and any attached documents may be confidential or legally privileged. It has been sent for the sole use of the intended recipient(s). If you are not an intended recipient, you are hereby notified that any unauthorized review, use, disclosure, dissemination, distribution, or copying of this communication or any attached documents, is strictly prohibited. If you have received this communication in error, please contact the sender by reply email and destroy all copies of the original message and any attached documents."

Appendix B

This section includes photographs taken during the experiment to identify the quantity of Sulphur in a sample of Nickel Sulphate Hexahydrate.



Figure 17,

Photograph of the X-ray Fluorescence Spectrometry analyser used during the experiment



Figure 18, Nickel Sulphate Hexahydrate samples taken from four individual consignments



Figure 19, Photograph of samples dissolving on a hotplate



Figure 20, Photograph of sample receptacles which are loaded into the X-ray Fluorescence Spectrometry analyser



Figure 21, Photograph of receptacles before loaded into the X-ray Fluorescence Spectrometry analyser

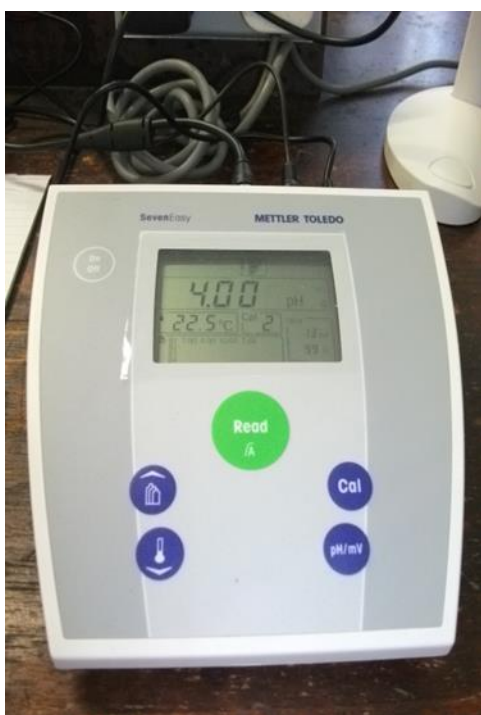


Figure 22, Photograph of Metler Toledo pH controller

Appendix C

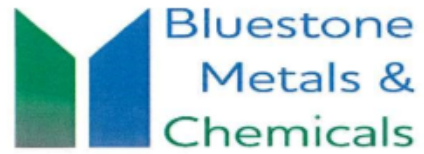
This section includes evidence of delivery weights, cost and, chemical composition of Nickel Sulphate Hexahydrate from the consignment: 23-143V which, samples were taken to analyse the quantity of Sulphur content.



Bulk FMW Discharged ex Containers

Date 25/4/24

54 | Page



Vale Europe Limited
Clydach Refinery
Swansea, SA6 5QR
UNITED KINGDOM

DATE 11-Apr-24
OUR REFERENCE NO S25.23.3809-16
YOUR REFERENCE NO 4510593984- Delivery 25/04/2024

PACKING LIST

COMMODITY Nickel Sulphate
QUANTITY 22,000
PACKING 1,000 kg bags
SHIPMENT BASIS DAP Swansea
COUNTRY OF DISPATCH United Kingdom
COUNTRY OF DESTINATION United Kingdom
SHIPMENT METHOD Truck

COMMODITY: Nickel Sulphate

LOT NUMBER	NO. OF BAGS	NO. OF PALLETS	KGS NET WEIGHT	KGS GROSS WEIGHT
23-143V	14	14	14,000	22,500
23-287V	8	8	8,000	

*In case you detect **Non Conformities** please always include them on the CMR and address it to ourselves so we can take action immediately.

*Bitte vermerken Sie im Falle einer **Reklamation** oder Abweichung einen Hinweis auf dem CMR und informieren Sie uns anschließend damit wir umgehend weitere Maßnahmen einleiten können.

* En caso de detectar **No Conformidades** por favor inclúyanlas siempre en el CMR e infórmennos para que podamos tomar medidas inmediatamente.

*Nel caso rilevaste delle **Non Conformità**, vi preghiamo di scriverle sempre sul CMR e di indirizzarle a noi via e-mail, in modo da poter agire immediatamente.

* Si vous constatez des **Non-Conformités** à la réception de nos produits, nous vous demandons à les inclure sur le CMR et nous les communiquer que nous puissions agir immédiatement.



Bluestone
Metals &
Chemicals

BLUESTONE METALS & CHEMICALS EUROPE S.L.U.
Carrer de Pascual Y Genís, 1 Planta 6
VALENCIA - SP 46002
Spain
+34 960 03 86 80
info@bluestonemc.com

External Ref No :	4510593984
Product Name :	NICKEL SULFATE
Lot Number :	23-143V

CERTIFICATE OF ANALYSIS

Parameters	Unit	Value
Ni	%	22.2960
Co	%	< 0.0001
Cu	%	< 0.0001
Fe	%	< 0.0001
Zn	%	< 0.0001
Mn	%	< 0.0001
Pb	%	< 0.0001
As	%	< 0.0001
Ca	%	0.0001

The information herein is believed to be reliable. However, no warranty express or implied, is made as to its accuracy or completeness and none is made as to the fitness of this material for any purpose. The manufacturer shall not be liable for damages to person or property resulting from its use. Nothing herein shall be construed as a recommendation for use in violation of any patent. Consult the SDS.



Bluestone Metals & Chemicals - Carrer de Pascual Y Genís, 1 Planta 6, SP 46002-Valencia, Spain | T +34960038680 | F +34960038681 | E info@bluestonemc.com

[illegible]

1. Shipper / Consignor / Sender Bluestone Metals & Chemicals Europe S.L. Calle Pascual y Genis 1, 6º 46002 VALENCIA SPAIN		2. Transport document number		
3. Page 1 of 1 Pages		4. Shipper's reference S25.23.3809-16		
5. Freight Forwarder's reference				
6. Consignee Vale Europe Ltd Clydach Refinery Swansea, SA6 5QR United Kingdom		7. Carrier (to be completed by the carrier)		
SHIPPER'S DECLARATION I hereby declare that the contents of this consignment are fully and accurately described below by the proper shipping name, and are classified, packaged, marked and labeled/placarded and are in all respects in proper condition for transport according to the applicable international and national governmental regulations.				
8. This shipment is within the limitations prescribed for: (Delete non-applicable) ROAD TRANSPORTATION WITHIN THE UK		9. Additional handling information		
10. Vessel / flight no. and date	11. Port / place of loading			
12. Port / place of discharge	13. Destination			
	Swansea, United Kingdom			
14. Shipping marks * Number and kind of packages; description of goods		Net Weight	Gross Weight (kg)	Cube (m3)
UN 3077 ENVIRONMENTALLY HAZARDOUS SUBSTANCE, SOLID, N.C.S. (NICKEL SULFATE), CLASS 9, PG III, F-A, S-F MARINE POLLUTANT 22 BAGS (13H4) OF 1000KG		22,000.00	22,500.00	
Emergency Phone Number: Antigifcentrum +32 (0) 70 245 245				
15. Container identification No. / vehicle registration No.		16. Seal number (s)		17. Container/vehicle size & type
				18. Tare (kg) 22,500.00
19. Total gross mass 22,500.00				
CONTAINER/VEHICLE PACKING CERTIFICATE I hereby declare that the goods described above have been packed/loaded into the container/vehicle identified above in accordance with the applicable provisions ** MUST BE COMPLETED AND SIGNED FOR ALL CONTAINER/VEHICLE LOADS BY PERSON RESPONSIBLE FOR PACKING/LOADING		21. RECEIVING ORGANISATION RECEIPT Received the above number of packages/containers/trailers in apparent good order and condition unless stated hereon: RECEIVING ORGANISATION REMARKS:		
20. Name of company Deny Logistics		Haulier's name 22. Name of company (OF SHIPPER PREPARING THIS NOTE) Bluestone Metals & Chemicals Europe S.L.		
Name / Status of declarant in case of emergency call:		Vehicle reg. no. Name / Status of declarant in case of emergency call: Werner van Dixhoorn +34 961 040 117		

VALE EUROPE LTD
CLYDACH
SWANSEA
SA6 5QR

GOODS IN TICKET

TRANSACTION No 53836

REGISTRATION KX65OWP Duplicate
SUPPLIER BLUESTONE-METAL-CHEMICAL

PRODUCT NICKEL SULPHATE

HAULIER S-K HAULAGE

DESTINATION VALE SWANSEA

ORDER No. 25/04/24
CONTAINER No. 449
NOTES
HAT/GLASSES YES

FIRST WEIGHT	38580	kg	040904	25/04/2024 12:41:4
SECOND WEIGHT	15820	kg	040907	25/04/2024 14:32:5
NET WEIGHT	22760	kg		

DRIVERS NAME PETERS

Operators Signature

Drivers Signature



A record of the weights used on this transaction will be kept for six months

Bluestone Sulphate
22 x 1T Bags
Lot No - 23-287V
Lot No - 23-143V
Del No - 525-23-3809-16
Bag date - 1-23

Bay 16
Powder
Warehouse.



Pro Forma Invoice

Date 19 APR 2024

Client's address:
TODINI UK LTD
2 Balfour Road
WEYBRIDGE, SURREY
Surrey KT13 8HD
UNITED KINGDOM

CGB09A202

Ref. client / Dd OFB 7 (01.2024) Apr /

Contract / Dd 2202076254 / 13 FEB 2024

Delivery / Dd 8235514 / 23 APR 2024

VAT number client GB398793903

Delivery address:
VALE EUROPE LIMITED
Clydach Refinery
Ynyspenllwch Road
SWANSEA SA6 5QR
UNITED KINGDOM

1017875

Contactperson: Kristel

Delivering plant: SUBS CSM BE04 Olen

Material Number	Product Custom tariff code	Quantity	Unit price (rounded to 2 dec.)	Total
29405135	Nickel Sulphate Electroless Grade, 22,3%, 1000 kg big bags 2833 2400 Country of Origin: BE	22,000.000 KG	4.30 USD/KG	94,695.73 USD
The exporter of the products covered by this document (BEREXBE0401574852) declares that, except where otherwise clearly indicated, these products are of EU preferential origin.				
VAT (code 42) (42) BE EXPORT - VAT EXEMPT				
Total weight		22,000.000 KG		
Total amount			94,695.73 USD	

Payment: 45 days end of month ,
Delivery: DAP SWANSEA by Trailer
Pro Forma Invoice for customs purpose only.
Remarks: EXPORTER OF RECORD BE 0401574852
Remarks: IMPORTER OF RECORD GB 398793903

IMPORTANT NOTICE

- Export formalities have to be done at a Belgian customs office
- Customs code and description of product : see commercial invoice
- ECS : message confirming exit of EU-territory to be addressed to :
customs@umicore.com

Coltalt Energy Products Olen
Umicore, SA/NV

Watertorenstraat 33 - B 2250 Olen - Belgium
Rue du Marais - 31 - Broekstraat
Tel (32-2) 227.76.36 - Fax (32-2) 227.79.30

Tel (32-14) 24.54.78 Fax (32-14) 24.57.56
B-1000 Bruxelles - Brussel
RCB/HRB 85382 - TVA/BTW BE0401574852



Packing List - Paklijst
Note de poids - Lieferschein
Consignment note - Vrachtbrief
Lettre de voiture - Frachtbrief

Del.nr. - leveringsnr. - n° de livraison - Liefer Nr. Date - Datum
8235514 23 APR 2024

Order - commande - Verkaufsbeleg Date - Datum
2202076254 13 FEB 2024

Y. ref. - U. ref. - vos réf. - Ihr Zeichen Date - Datum

OFB 7 (01.2024) Apr

Carrier - vervoerder - expéditeur - Spedition Transport-ID

DSV ROAD NV/SA

Agent

Correspondent - correspondant - Ansprechpartner Tel.

Kristel

Plant - fabriek - usine - Werk

SUBS CSM BE04 Olen

Destination - bestemming - Lieferanschrift

1017875

VALE EUROPE LIMITED

Clydach Refinery

Ynyspennwlch Road

SWANSEA SA6 5QR

Client - klant - Kunde

CGB09A202

TODINI UK LTD

2 Balfour Road

WEYBRIDGE, SURREY KT13 8HD

UNITED KINGDOM

Shipping marks 2202076254

Country

United Kingdom

Unloading point - reference - vessel - closing - destination - agent

Losplaats - referentie - schip - sluiting - bestemming - agent

Point de décharge - référence - bateau/embarcadere - destination - age

Abladestelle - Verweis - Schiff - Schliessung - Bestimmung - Agen

F201 UK Customs Agent Name:Jeavons Eurotir Ltd-

customs@jeavonseurotir.co.uk-tel 441212010064.

Importer EORI Nr: GB 398793903 cc

entrte@todini.com/delivery to

book in adv./Contact person :Mr.Phil

BOWDEN,phil.bowden@vale.com

Mat.nr. N° Article	Customs Tariff code - douanetariefscode - Code du tarif douanier - Zolltarife Code Description - beschrijving - désignation - Bezeichnung	Quantity Hoeveelheid Quantité Menge	Net weight Nettogewicht Poids net Netto Gewicht (kg)	Tarra Tara Tare Tara (kg)	Gross weight Bruto gewicht Poids brut Bruto Gewicht (kg)
29405135	2833 2400 Nickel Sulphate Electroless Grade, 22,3%, 1000 kg big bags UN 3077 MILIEUGEVARAARLIJKE VASTE STOF, N.E.G. (Nikkelsulfaat Hexahydraat), 9, III, (-) UN 3077 MATIÈRE DANGEREUSE DU POINT DE VUE DE L'ENVIRONNEMENT, SOLIDE, N.S.A. (Sulfate de Nickel Hexahydrate), 9, III, (-) UN 3077 ENVIRONMENTALLY HAZARDOUS SUBSTANCE, SOLID, N.O.S. (Nickel Sulphate Hexahydrate) 9, III, MARINE POLLUTANT Segregation Group: Not Applicable EMS n° F-A/S-F	22	22,000.000	66.000	22,066.000
	Palet - Palette:	22		440.000	440.000
	Totals - totaal - total - Gesamt		22,000.000	506.000	22,506.000

Incoterms: DAP, Swansea

Comments - bemerkingen - commentaires - Bemerkungen

For receipt - voor ontvangst
pour réception - für Empfänger

transporter - vervoerder
transporteur - Fahrer

Cobalt Energy Products Olen
Umicore, SA/NV

Watertorenstraat 33 - B 2250 Olen - Belgium
Rue du Marais - 31 - Brockstraat
Tel (32-2) 227.76.36 - Fax (32-2) 227.79.30

Tel (32-14) 24.54.78 Fax (32-14) 24.57.56
B-1000 Bruxelles - Brussel
RCB/HRB 85382 - TVA/BTW BE0401574852

MULTI-MODAL DANGEROUS GOODS FORM

This form may be used as a dangerous goods declaration as it meets the requirements of SOLAS 74, chapter VII, regulation 4; MARPOL 73/78, Annex III, regulation 4 and IMDG Code

1 Shipper/Consignor/Sender Umicore, SA/NV Industrieweg 34 2280 Grobbendonk BE		2 Transport document number 8235514	
		3 Page 1 of pages 1	4 Shipper's reference 2202076254
6 Consignee VALE EUROPE LIMITED Clydach Refinery Ynyspenllwch Road SWANSEA SA6 5QR UNITED KINGDOM		7 Carrier (to be completed by the carrier)	
8 This shipment is within the limitations prescribed for: (Delete non-applicable) PASSENGER AND CARGO AIRCRAFT		9 Additional handling information 24 hours emergency number: Carechem24/Umicore Region Europe: +32 3 213 15 70	
10 Vessel/flight no. and date	11 Port/place of loading Grobbendonk/BE/		
12 Port/place of discharge	13 Destination Swansea/GB		
14 Shipping marks * Number and kind of packages; description of goods Gross mass (kg) Net mass (kg) Cube (m³)			
22 BIG BAG 1000 KG 22.066,000 22.000,000 Nickel Sulphate Electroless Grade, 22,3%, 1000 kg big bags UN 3077 MILIEUGEVAARLIJKE VASTE STOF, N.E.G. (Nikkelsulfaat Hexahydraat), 9, III, (-) UN 3077 MATIERE DANGEREUSE DU POINT DE VUE DE L'ENVIRONNEMENT, SOLIDE, N.S.A. (Sulfate de Nickel Hexahydrate), 9, III, (-) UN 3077 ENVIRONMENTALLY HAZARDOUS SUBSTANCE, SOLID, N.O.S. (Nickel Sulphate Hexahydrate) 9, III, MARINE POLLUTANT Segregation Group: Not Applicable EMS n° F-A/S-F SDS : 300000002057			
15 Container identification No./ vehicle registration No.	16 Seal number(s)	17 Container/Vehicle size & type ☐ Open ☐ Closed size:	18 Tare mass (kg) 19 Total gross (including tare) (kg)
CONTAINER/VEHICLE PACKING CERTIFICATE I hereby declare that the goods described above have been packed/ loaded into the container/vehicle identified above in accordance with the applicable provisions.** MUST BE COMPLETED AND SIGNED FOR ALL CONTAINER/VEHICLE LOADS BY PERSON RESPONSIBLE FOR PACKING/LOADING.		21 RECEIVING ORGANIZATION RECEIPT Received the above number of packages/containers/trailers in apparent good order and condition, unless stated hereon: RECEIVING ORGANIZATION REMARKS:	
20 Name of company Umicore, SA/NV	Haulier's name BATIM Vehicle reg. no. 23.04.24	22 Name of company (OF SHIPPER PREPARING THIS NOTE) Umicore, SA/NV	
Name/status of declarant Kimberly Willemsen	Signature and date 23.04.24	Name/status of declarant Kimberly Willemsen	
Place and date Grobbendonk/BE/	DRIVER'S SIGNATURE	Place and date Grobbendonk/BE/	
Signature of declarant		Signature of declarant	

* DANGEROUS GOODS:
You must specify: UN No., proper shipping name, hazard class, Packing groups (where assigned), MARINE POLLUTANT and observe the mandatory requirements under the applicable national and international governmental regulations. For the purposes of the IMDG Code see 5.4.1.1.
** For the purposes of the IMDG Code see 5.4.2.

See overleaf for documentary aspects.

2

Exemplaire pour le destinataire • Exemplaar voor geadresseerde • Exemplar für Empfänger

LETTRE DE VOITURE • VRACHTBRIEF • FRACHTBRIEF



B

N°

Nr

FEB

210958810

Le transport est soumis, nonobstant toute clause contraire, à la Convention CMR.
 De vervoer is, ongeacht enig tegenstrijdig beding, onderworpen aan het CMR-Verdrag.
 Trotz gegenteiliger Abmachung unterliegt diese Befrachtung den Bestimmungen des CMR-Übereinkommens.

1 Expéditeur ou commissionnaire (nom, adresse, État) • Afzender of commissiër (naam, adres, Staat) • Absender oder Spediteur (Name, Anschrift, Staat) Umicore, SA/NV Industrieweg 34 2280 Grobbendonk Umicore Grobbendonk/BE/	5 Transporteur principal (nom, adresse, État, n° de licence, plaque(s) d'immatriculation) • Hoofdvervoerder (naam, adres, Staat, vergoeding, plaatnummer(s)) • Hauptfrachtführer (Name, Anschrift, Staat, Genehmigungs-nr., amtliche(s) Kennzeichen) DSV ROAD NV/SA Transportcentrum L.A.R 8930 Rekken	
2 Destinataire (nom, adresse, État) • Geadresseerde (naam, adres, Staat) • Empfänger (Name, Anschrift, Staat) VALE EUROPE LIMITED Clydach Refinery Ynyspenllwch Road SA6 5QR Swansea	6 Transporteur sous-traitant (nom, adresse, État, n° de licence, plaque(s) d'immatriculation) • Ondervervoerder (naam, adres, Staat, vergoeding, plaatnummer(s)) • Unterfrachtführer (Name, Anschrift, Staat, Genehmigungs-nr., amtliche(s) Kennzeichen) BATIM TRANSPORT MIĘDZYNARODOWY I SPEDYTOR ul. Prymasa Stefana Wyszyńskiego 9 33-340 Stary Sącz Poland www.batim.com.pl	
3 Prise en charge de la marchandise (lieu, État, date, heure) • Inontvangstneming van de goederen (plaats, Staat, datum, uur) • Warenabnahme (Ort, Staat, Datum, Uhr) Grobbendonk Umicore, SA/NV Grobbendonk/BE/	7 Transporteur successeur (nom, adresse, État, n° de licence, plaque(s) d'immatriculation) • Opvolgende vervoerder (naam, adres, Staat, vergoeding, plaatnummer(s)) • Nachfolgender Frachtführer (Name, Anschrift, Staat, Genehmigungs-nr., amtliche(s) Kennzeichen) KNUUBZC / KN 025PC	
4 Livraison (lieu, État, date, heure) • Aflevering (plaats, Staat, datum, uur) • Lieferung (Ort, Staat, Datum, Uhr) Swansea	9 Réserves du transporteur lors de la prise en charge de la marchandise • Voorbehoud van de vervoerder bij de ontvangstneming van de goederen • Vorbehalte des Frachtführers bei der Warenabnahme	
8 Frais effectués au transport • Transportgebonden kosten • Transportgebundene Kosten ☐ Temps d'attente • Wachtijden • Wartezeiten ☐		
10 Marchandises transportées (nature, nombre, poids brut ou net, emballage, marque et n°s, ...) • Vervoerde goederen (aard, aantal, bruto- of nettogewicht, verpakking, merken en nrs, ...) • Beförderte Güter (Art, Anzahl, Brutto- oder Nettogewicht, Verpackung, Kennzeichen und Nrs, ...) NICKEL SULPHATE EN GRADE, BIG BAGS 1000KG 22.000,000KG 22.066,000 SDS : 300000002057 22,000 Pieces UN 3077 MILIEUGEVAARLIJKE VASTE STOF, N.E.G. (Nikkelsulfaat Hexahydra 9, III, (-) UN 3077 MATIÈRE DANGEREUSE DU POINT DE VUE DE L'ENVIRONNEMENT, SOLIDE N.S.A. (Sulfate de Nickel Hexahydrate), 9, III, (-) 22 Palets 440,000 Total weight 22.506,000 KG		
11 Documents annexes transmis par l'expéditeur • Door de afzender bezorgde aanvullende documenten • vom Absender zusätzlich übermittelte Dokumente 24 hours emergency number: Carechem24/Umicore Region Europe: +32 3 213 15 70	13 Instructions de l'expéditeur • Instructies van de afzender • Anweisungen des Absenders ☐ Contre remboursement • Onder rembours • gegen Nachnahme ☐ Reprise palettes vides • Terugneme lege palletten • Zurücknahme der leeren Ladepaletten ☐ Delivery number: 8235514 Ref. number : 2202076254 Ref. Customer : OFB 7	
12 Lieu et date d'établissement • Plaats en datum van opmaak • Aufstellungsart und Datum 2280 Grobbendonk Umicore 10.04.2024		
14 Umicore, SA/NV Industrieweg 34 2280 Grobbendonk Grobbendonk/BE/ Kimberly Willemssen	15 BATIM TRANSPORT MIĘDZYNARODOWY I SPEDYTOR ul. Prymasa Stefana Wyszyńskiego 9 33-340 Stary Sącz Poland www.batim.com.pl	16

• Autokopie worden op grond van het M.B. van 23/05/2014 • Reproduction interdite sur base du M.B. du 23/05/2014 • Nachdruck verboten gemäß ministeriellem Erlass vom 23/05/2014

E-mail: fact@carechem.be
 http://www.carechem.be

02 485 80 00
 02 428 00 96



EUROPESE GEMEENSCHAP

UITVOERGELEIDE DOCUMENT	Afzender/Expéditeur (2) Nr. BE0401574852 UN RUE DU MARAIS 31 1000 BRUXELLES BE		SOORT AANKOMST (1) EU A Indiv. ABO (S32)		MRN 24BEE0000025218508 	
	Gedestineerde (8) M. geen VALE EUROPE LIMITED CLYDACH REFINERY YNYSPENLACH SA65QR SWANSEA GB		Formulieren (3) 1 1 S		Aang. coll. (S00)	
	Aangever/Verleganwoordgar (14) Nr. BE0404527216 GERLACH & CO SCHOUWENSS TRAAAT 7 2030 ANTWERPEN		Artikelen (5) 22		Totaal Coll. (6) 22	
	Identiteit van het vervoermiddel bij vertrek (18) KN285PC		Referentienummer (7) GB-966924-2202076254-8235514		Datum van afgifte: 22/04/2024 Douanekantoor: BSHSS216000	
	Vervoerswijze aan de grens (25) 3		Plaats van de goederen (30) BEGDBHPC0404004		Code betalingswijze vervoerskosten (S28) a 32	
	Kantoor van uitgang (29) FR620001		Verleganwoordgar inzien sum. aangifte (14b) Nr.		Code L. varz. uitv. (15) a GB	
	Merk en omschrijving van de goederen (31) 22 PK 29405135		Buitomassa (kg) (35) 22506,000		Code L. bestemming (17) a GB	
	Merk en nummers - Container(s) n(s) - Aantal en soort 22 PK 29405135		Verzegelingsnummer (S28)		Code(s) land(en) die deel uitmaken van het vervoerstraject (S13)	
	Artik. (32)		Aantal en soort coll., serial stuks, merken en nummers van de coll. (31/1)		Omschrijving van de goederen (31/2)	
	Afzender/Expéditeur (2)		Gedestineerde (8)		Gedestineerde (23)	
Identiteit van het vervoermiddel bij vertrek (18)		Summiere aangifte/voorafgaand document (40)		Nummer(s) van de carline(s) (31/3)		
Uniek referentienummer van de zending (7)		Ragaling (37)		Land van uitv. (15a)		
Voorgeslede slukken/verl. (44/1)		Land van uitv. (15a)		L. bestemm. (17a)		
Bijzondere vermeldingen (44/2)		Soort aangifte (1)		Statistische waarde (46)		
LNDG (44/4)		Code betalingswijze vervoerskosten (S28)		Nietomassa (kg) (38)		
1		22 PK 29405135		NIKKELSULFATEN		
KN285PC		28332400 00 0000 0000		E-XXX NIKEL 00/00/0000		
GB-966924-2202076254-8235514		10 00		22		
MRN 24BEE0000025218508		GB		22506,000		
BE0401574852		86525,46		22506,000		
ALGEMOET BE0404527216						

E CONTROLE DOOR HET KANTOOR VAN VERZENDING/UITVOER

Uitslag:

Aangebrachte verzegeling: Aantal:
merken:

Termijn uitbreide datum:

CONTROLE DOOR HET KANTOOR VAN UITGANG (K)

Datum van aankomst:

Controle van de verzegeling:

Opmerkingen:

VALE EUROPE LTD
CLYDACH
SWANSEA
SA6 5QR

GOODS IN TICKET

TRANSACTION No 53835

REGISTRATION

KN1473C

Duplicate

SUPPLIER

UMICORE

PRODUCT

NICKEL SULPHATE

HAULIER

DSV TRANSPORT

DESTINATION

VALE SWANSEA

ORDER No.
CONTAINER No.
NOTES
HAT/GLASSES

25/04/24
KN285PC

YES

FIRST WEIGHT
SECOND WEIGHT
NET WEIGHT

38160 kg
15660 kg
22500 kg

040903
040906

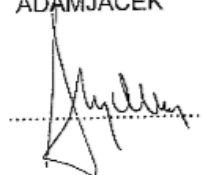
25/04/2024 12:34:3
25/04/2024 13:41:1

DRIVERS NAME

ADAMJACEK

Operators Signature

Drivers Signature



A record of the weights used on this transaction will be kept for six months

UMICORE SULPHATE
22 x 1 T BAGS
Delivery No - 8235514
LOT No - 2940502699
Bag date - 01/23
Bag 17 Powder Warehouse.

Appendix D

This section includes evidence of delivery weights, cost and, chemical composition of Nickel Sulphate Hexahydrate from the consignment: 2940502697 which, samples were taken to analyse the quantity of Sulphur content.

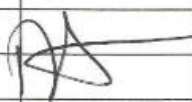


VALE EUROPE LIMITED
CLYDACH REFINERY

Nº 9274

Bulk FMW Discharged ex Containers

Date 14/3/24

Vessel	Container No.	Lot No.	Silo used	Actual Gross Weight	Advised Gross Weight	Variance
DSV	L21 ERL TYPE 657128				22540	
TONDINI	UMICORE SULPHATE 22 x 1 TONNE BAG LOT N° - 2940502697 DEL N° - 8233020 BAG DATE 4/23 BAY 6 - POWDER WAREHOUSE					
	L21 SUV SUL394				22540	
TONDINI	UMICORE SULPHATE 22 x 1 TONNE BAG LOT N° - 2940502697 DEL N° - 8233021 BAG DATE 4/23 BAY 11 POWDER WAREHOUSE					
						



Digitale en fysieke verificatie van de afzender
Handtekening en stempel van de afzender of commissionair
Unterschrift und Stempel des Absenders oder Spediteurs

Digitale en fysieke verificatie van de afzender
Handtekening en stempel van de werkelijke verzender
Unterschrift und Stempel des tatsächlichen Fachführers

Digitale en fysieke verificatie van de afzender
Handtekening, datum en stempel van de geadresseerde
Unterschrift, Datum und Stempel des Empfängers



VALE EUROPE LTD
CLYDACH
SWANSEA
SA6 5QR

GOODS IN TICKET

TRANSACTION No 53470

REGISTRATION LL21ERL

SUPPLIER UMICORE

PRODUCT NICKEL SULPHATE BAGS

HAULIER DSV TRANSPORT

DESTINATION VALE SWANSEA

ORDER No. 14/03/24
CONTAINER No. TIPE6571286
NOTES
HAT/GLASSES YES

FIRST WEIGHT	38520	kg	040188	14/03/2024 12:33:2
SECOND WEIGHT	15980	kg	040191	14/03/2024 13:37:1
NET WEIGHT	22540	kg		

DRIVERS NAME BELLINGHAM

Operators Signature

Drivers Signature

A record of the weights used on this transaction will be kept for six months

TONDINI UMICORE SULPHATE

22 x 1 TONNE BAGS

LOT No - 2940502697

DEL No - 8233020

BAG DATE - 4/23

LOCATION - BAY 6 POWDER WAREHOUSE



Packing List - Paklijst
Note de poids - Lieferschein
Consignment note - Vrachtbrief
Lettre de voiture - Frachtbrief

Del.nr. - leveringsnr. - n° de livraison - Liefer Nr. Date - Datum
8233021 12 MAR 2024

Order - commande - Verkaufsbeleg Date - Datum
2202076246 13 FEB 2024

Y. ref. - U. ref. - vos réf. - Ihr Zeichen Date - Datum

OFB 7 (01.2024) Mar

Carrier - vervoerder - expéditeur - Spedition Transport-ID

DSV ROAD NV/SA

Agent

Correspondant - correspondant - Ansprechpartner Tel.

Jana

Plant - fabriek - usine - Werk

SUBS CSM BE04 Olen

Destination - bestemming - Lieferanschrift

1017875

VALE EUROPE LIMITED

Clydach Refinery

Ynyspenllwch Road

SWANSEA SA6 5QR

Client - klant - Kunde

CGB09A202

TODINI UK LTD

2 Balfour Road

WEYBRIDGE, SURREY KT13 8HD

UNITED KINGDOM

Shipping marks 2202076246

Country

United Kingdom

Unloading point - reference - vessel - closing - destination - agent

Losplaats - referentie - schip - stuiting - bestemming - agent

Point de décharge - référence - bateau/embarcadere - destination - age

Abladestelle - Verweis - Schiff - Schliessung - Bestimmung - Agen

F201 UK Customs Agent Name: Jeavons Eurottr Ltd

customs@jeavonseurottr.co.uk-tel 441212010064.

Importer EORI Nr: GB 398793903 cc

entrance@todini.com/delivery to

book in adv./Contact person : Mr. Phil

BOWDEN, phil.bowden@vale.com

Mat.nr. N° Article	Customs Tariff code - douanetariefscode - Code du tarif douanier - Zolltarife Code Description - beschrijving - désignation - Bezeichnung	Quantity Hoeveelheid Quantité Menge	Net weight Nettogewicht Poids net Netto Gewicht (kg)	Tariff Tarifa Tara Tara (kg)	Gross weight Bruto gewicht Poids brut Bruto Gewicht (kg)
29405135	2833 2400 Nickel Sulphate Electroless Grade, 22,3%, 1000 kg big bags UN 3077 MILIEUGEVAARLIJKE VASTE STOF, N.E.G. (Nikkelsulfaat Hexahydraat), 9, III, (-) UN 3077 MATIÈRE DANGEREUSE DU POINT DE VUE DE L'ENVIRONNEMENT, SOLIDE, N.S.A. (Sulfate de Nickel Hexahydrate), 9, III, (-) UN 3077 ENVIRONMENTALLY HAZARDOUS SUBSTANCE, SOLID, N.O.S. (Nickel Sulphate Hexahydrate) 9, III, MARINE POLLUTANT Segregation Group: Not Applicable EMS n° F-A/S-F	22	22,000.000	66.000	22,066.000
	Palet - Palette:	22		440.000	440.000
	Totals - totaal - total - Gesamt		22,000.000	506.000	22,506.000

Incoterms: DAP, Swansea

Comments - bemerkingen - commentaires - Bemerkungen

For receipt - voor ontvangst
pour réception - für Empfänger

transporter - vervoerder
transporteur - Fahrer

Cobalt Energy Products Olen
Umicore, SA/NV

Waterloosestraat 33 - B 2250 Olen - Belgium
Rue du Marais - 31 - Broekstraat
Tel (32-2) 227.76.36 - Fax (32-2) 227.79.30

Tel (32-14) 24.54.78 Fax (32-14) 24.57.56
B-1000 Bruxelles - Brussel
RCB/HRB 85382 - TVA/BTW BE0401574852

3

Exemplaire pour le transporteur • Exemplaar voor vervoerder • Exemplar für Frachtführer

LETTRE DE VOITURE • VRACHTBRIEF • FRACHTBRIEF



B

N° FEB

110055813

Ce transport est soumis, nonobstant toute clause contraire, à la Convention CMR.
De vervoer is, ongeacht enig tegengesteld beding, onderworpen aan het CMR Verdrag.
Trotz gegenteiliger Abmachung unterliegt diese Befrachtung den Bestimmungen des CMR Übereinkommens.

1 Expéditeur ou commissionnaire (nom, adresse, Etat) • Abzender oder Spediteur (Name, Anschrift, Staat) Umicore, SA/NV Industrieweg 34 2280 Grobbendonk Grobbendonk/BE/	5 Transporteur principal (nom, adresse, Etat, n° de licence, plaque(s) d'immatriculation) Hoofdvervoerder (naam, adres, Staat, vergoedingsnr., plakkennummer(s)) Hauptfrachtführer (Name, Anschrift, Staat, Genehmigungsnummer, amtliches Kennzeichen) DSV ROAD NV/SA Transportcentrum L.A.R. 8930 Rekken
2 Destinataire (nom, adresse, Etat) • Geadresseerde (naam, adres, Staat) Empfänger (Name, Anschrift, Staat) VALE EUROPE LIMITED Clydach Refinery Ynyspenllwch Road SA6 5QR Swansea	MDQ Koffieweg 2 5380 Zeelburg JUL 394 206150V
3 Prise en charge de la marchandise (date, Etat, date, heure) • Warenontvangst (Off. Staat, Datum, Uhr) Grobbendonk Grobbendonk/BE/	6 Transporteur sous-traitant (nom, adresse, Etat, n° de licence, plaque(s) d'immatriculation) Ondervervoerder (naam, adres, Staat, vergoedingsnr., plakkennummer(s)) Unterfrachtführer (Name, Anschrift, Staat, Genehmigungsnummer, amtliches Kennzeichen) ZLP Tras MED 2 TRANS Tlancakukelau 173
4 Livraison (date, Etat, date, heure) • Aflevering (datum, Staat, datum, uur) Lieferung (Off. Staat, Datum, Uhr) Swansea	8400 005 fad
8 Frais affranchissement • Transportgebühren • Transportgebühren ☐ Taxes d'entente • Wachbilden • Wachbilden ☐ 8440 100	9 Réserve du transporteur (date, Etat, date, heure) • Voorbehoud van de vervoerder (datum, Staat, datum, uur) by de ontvangstneming van de goederen • Valbehoede des Frachtführers (bei der Warenabnahme)
10 Marchandises transportées (nature, nombre, poids brut ou net, emballage, marques et n°s, ...) • Vervoerde goederen (aart, aantal, bruto- of nettogewicht, verpakking, kenmerken en nrs, ...) Beforderte Güter (Art, Anzahl, Brutto- oder Nettogewicht, Verpackung, Kennzeichen und Nrs, ...) NICKEL SULPHATE EN GRADE, BIG BAGS 1000KG 22.000,000KG 22.066,000 SDS : 300000002057 22,000 Pieces UN 3077 MILIEUGEVAARLIJKE VASTE STOF, N.E.G. (Nikkelsulfaat Hexahydrat) 9, III, (-) UN 3077 MATIÈRE DANGEREUSE DU POINT DE VUE DE L'ENVIRONNEMENT, SOLIDE N.S.A. (Sulfate de Nickel Hexahydrate), 9, III, (-) 22 Palets 440,000 Total weight 22.506,000 KG Referentie: 2202076246 Seal: NDQ 031701	
11 Documents annexes transmis par l'expéditeur • Door de afzender bezorgde aanvullende documenten • Durch den Absender beigegebene Dokumente Carechem24/Umicore Region Europe: +32 3 213 15 70	13 Instructions de l'expéditeur • Instrucciones van de afzender • Anweisungen des Absenders ☐ Contre-remboursement • Contar rimbours • gegen Nachnahme ☐ Reprise palettes vides • Torngame lege paletten • Zurücknahme der leeren Ladepaletten Delivery number: 8233021 Ref. number : 2202076246 Ref. Customer : OFB 7
12 Lieu et date d'établissement • Plaats en datum van opmaak • Aufstellungsort und Datum 2280 Grobbendonk 11.03.2024	14 Umicore, SA/NV Industrieweg 34 2280 Grobbendonk Grobbendonk/BE/ Siebe Geubelmans
15 2102117	16 DA

Reproduced with permission from the CMR Convention • Reproduktion mit Erlaubnis der CMR Konvention • Reproduktion mit Erlaubnis der CMR Konvention

E-mail: info@cmr-convention.eu
http://www.cmr-convention.eu

VALE EUROPE LTD CLYDACH SWANSEA SA6 5QR	GOODS IN TICKET	TRANSACTION No	53473
REGISTRATION	LS21SUV		
SUPPLIER	UMICORE		
PRODUCT	NICKEL SULPHATE BAGS		
HAULIER	NDQ		
DESTINATION	VALE SWANSEA		
ORDER No.	14/03/24		
CONTAINER No.	JUL394		
NOTES			
HAT/GLASSES	YES		
FIRST WEIGHT	37600 kg	040189	14/03/2024 12:38:0
SECOND WEIGHT	15060 kg	040194	14/03/2024 14:27:2
NET WEIGHT	22540 kg		

DRIVERS NAME MEHANOVIC

Operators Signature

Drivers Signature

A record of the weights used on this transaction will be kept for six months

TONDINI UMICORE SULPHATE
 22 x 1 TONNE BAGS
 LOT NO 2940502697
 DEL NO - 8233021
 BAG DATE - 4/23
 LOCATION - BAY 11 Powder Warehouse

Appendix E

This section includes evidence of delivery weights, cost and, chemical composition of Nickel Sulphate Hexahydrate from the consignment: 23-399V which, samples were taken to analyse the quantity of Sulphur content.

1

INSTRUCTION:

OUTBOUND

Client	BLUESVAL(ES)
Consignor	BLUESVAL(ES)
Reference (for invoice)	S25.23.3809-5
Condition	DAP
Carrier	S&K
SWANSEA	

Company name	JPS
Address	Ashcroft Road, Knowsley Industrial Park
Postal code	L33 7TS
City	Kirkby
Country	UK
Collection / ship date	Please check with JPS
Collection time	
Loading reference	
Total # Pallets	22

Company name	Vale Europe Limited
Address	Clydach Refinery
Postal code	S46 5QR
City	Swansea
Country	United Kingdom
Delivery date	29-02-2024
Delivery time	12:30
Unloading reference	S25.23.3809-5

Company name	JPS
Address	Ashcroft Road, Knowsley Industrial Park
Postal code	L33 7TS
City	Kirkby
Country	UK
Collection / ship date	Please check with JPS
Collection time	
Loading reference	
Total # Pallets	22

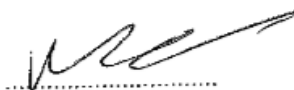
Company name	JPS
Address	Ashcroft Road, Knowsley Industrial Park
Postal code	L33 7TS
City	Kirkby
Country	UK
Collection / ship date	Please check with JPS
Collection time	
Loading reference	
Total # Pallets	22

Company name	Vale Europe Limited
Address	Clydach Refinery
Postal code	S46 5QR
City	Swansea
Country	United Kingdom
Delivery date	29-02-2024
Delivery time	12:30
Unloading reference	S25.23.3809-5

Article	Description	Batch	Customs	Kg Net	Kg Gross	# pallets	HS-code	UW	ADR	PG
NISO - 10000 - 1000000000	NICKEL SULFATE	13-4000V	Free	10,000	22,500	14	2833.24	3077	9	III
NISO - 10000 - 1000000000	NICKEL SULFATE	23-3995V	Free	12,000	22,500	8	2833.24	3077	9	III

VALE EUROPE LTD CLYDACH SWANSEA SA6 5QR	GOODS IN TICKET	TRANSACTION No	53326
REGISTRATION	GH16LUJ		
SUPPLIER	JPS		
PRODUCT	NICKEL SULPHATE BAGS		
HAULIER	S-K HAULAGE		
DESTINATION	VALE SWANSEA		
ORDER No.	29/02/24		
CONTAINER No.	T315		
NOTES			
HAT/GLASSES	YES		
FIRST WEIGHT	38860 kg	039901	29/02/2024 12:33:5
SECOND WEIGHT	16120 kg	039909	29/02/2024 13:41:3
NET WEIGHT	22740 kg		

DRIVERS NAME MARTIN

Operators Signature Drivers Signature 

A record of the weights used on this transaction will be kept for six months

BLUESTONE SULPHATE 22x11 BAGS

BAY 2 POWDER WAREHOUSE

LOT N° 23-399V

BAG DATE.
02/24

DEL N° S25.23,3809-5

Appendix F

This section includes evidence of delivery weights, cost and, chemical composition of Nickel Sulphate Hexahydrate from the consignment: 2940502620 which, samples were taken to analyse the quantity of Sulphur content.



Bulk FMW Discharged ex Containers

Date 21/12/23

Vessel	Container No.	Lot No.	Silo used	Actual Gross Weight	Advised Gross Weight	Variance
0969 ZYZ	DSVA3803421				22,600	
TONDI NI UNICORE SULPHATE 22 X 1 TONNE BAGS BAY 7 POWDER WAREHOUSE						
LOT N°- 2940502620						
DEL N°- 8228742						
BAG DATE- 09/22						



Delivered by DSV Road Limited



RK2UK-AB074

Deliver
VALE EUROPE LIMITED
YNYPENLLWCH ROAD
SWANSEA
SA6 5QR

Phone 1792 8 42 501
Delv. 2023-12-21
Delv. Time 12:30-12:30
Ref 8228742-2202067826

Contact ANDREW HORTON
Phone +44 (0) 1255 244088

Page 2(3)

Shipper
UMICORE SA
UM SA
BE-1000 BRUXELLES
BELGIUM

CMR-CONSIGN. NOTE

Consignee
VALE EUROPE LIMITED
YNYPENLLWCH ROAD
SWANSEA
SA6 5QR

Date 2023-12-20
Reference BEUK10-2781 RK2UK-AB074
Terms DELIVERED AT PLACE
DAP
SWANSEA
Originating member BEUK10

Coll. 2023-12-19/07:30-15:00
Unit.no DSV3803421

Haulier -
Reg.no. -

UN 3077, ENVIRONMENTALLY HAZARDOUS SUBSTANCE, SOLID, N.O.S., (ENVIRONMENTALLY HAZARDOUS SUBSTANCE), SOLID, (-), 20
CLL, 20000.0 KGS
UN 3077, ENVIRONMENTALLY HAZARDOUS SUBSTANCE, SOLID, N.O.S., (ENVIRONMENTALLY HAZARDOUS SUBSTANCE), SOLID, (-), 2
CLL, 2000.0 KGS
CHRIS.BORGES@UMICORE.COM
CHRIS BORGES

Marks & Nos	No. of	Pkg.	Contents	Weight kg	Volume
2202067826	22	PLL	NON-STACKABLE / NICK	GW 24000	22.000 CBM 13.6 LDM

EL SULPHATE ELECTROL
ESS GRADE 22.3%, 10
00 KG BIG BAGS

UN 3077, ENVIRONMENTALLY HAZARDOUS SUBSTANCE, SOLID, N.O.S., (ENVIRONMENTALLY HAZARDOUS SUBSTANCE), SOLID, (-), 20
CLL, 20000.0 KGS
UN 3077, ENVIRONMENTALLY HAZARDOUS SUBSTANCE, SOLID, N.O.S., (ENVIRONMENTALLY HAZARDOUS SUBSTANCE), SOLID, (-), 2
CLL, 2000.0 KGS

Notwithstanding anything to the contrary herein, the carriage is subject to the CMR Convention as adopted by the Executive Order 1986-09-09 No. 602 on contract in International carriage by road.

PIN 8272 F201 UK CUSTOMS AGENT NAME: DSV-E MAIL: SOPHIE.TURNER@UK.DSV.COM / EORI NR :
GB778655171000/DELIVERY TO BE BOOKED IN ADVANCED / CONTACT PERSON : MR. PHIL BOWDEN ,

Marks & Nos	No. of	Pkg.	Contents	Weight	KG	Volume
2202067826	22	PLL	NON-STACKABLE / NICK	GW	24000	22.000 CBM
				NW	22506	
				EW	24000	13.6 LDM
Returnable items	12	SPAN				

www.dsv.com



Delivered by DSV Road Limited



RK2UK-AB074

Deliver
VALE EUROPE LIMITED
YNYSPENLLWCH ROAD
SWANSEA
SA6 5QR

Phone 1792 8 42 501
Delv. 2023-12-21
Delv. Time 12:30-12:30

Contact ANDREW HORTON
Phone +44 (0) 1255 244088

Page 3(3)

Shipper
UMICORE SA
UM SA
BE-1000 BRUXELLES
BELGIUM

HAZARDOUS GOODS SPECIFICATION

Consignee
VALE EUROPE LIMITED
YNYSPENLLWCH ROAD
SWANSEA
SA6 5QR

Date 2023-12-20
Reference BEUK10-2781 RK2UK-AB074
Terms DELIVERED AT PLACE
DAP
SWANSEA

Hazardous Goods Specification

UN 3077, ENVIRONMENTALLY HAZARDOUS SUBSTANCE, SOLID, N.O.S., (ENVIRONMENTALLY HAZARDOUS SUBSTANCE), SOLID, (-), 20
CLL, 20000.0 KGS
UN 3077, ENVIRONMENTALLY HAZARDOUS SUBSTANCE, SOLID, N.O.S., (ENVIRONMENTALLY HAZARDOUS SUBSTANCE), SOLID, (-), 2
CLL, 2000.0 KGS
UN 3077, ENVIRONMENTALLY HAZARDOUS SUBSTANCE, SOLID, N.O.S., (ENVIRONMENTALLY HAZARDOUS SUBSTANCE), SOLID, (-), 20
CLL, 20000.0 KGS
UN 3077, ENVIRONMENTALLY HAZARDOUS SUBSTANCE, SOLID, N.O.S., (ENVIRONMENTALLY HAZARDOUS SUBSTANCE), SOLID, (-), 2
CLL, 2000.0 KGS



Packing List - Paklijst
Note de poids - Lieferschein
Consignment note - Vrachtbrief
Lettre de voiture - Frachtbrief

Del.nr. - leveringnr. - n° de livraison - Liefer Nr. Date - Datum
8228742 19 DEC 2023

Order - commande - Verkaufsbeleg Date - Datum
2202067826 15 DEC 2022

Y. ref. - U. ref. - vos réf. - Ihr Zeichen Date - Datum

OFK 58 (12.2022)

Carrier - vervoerder - expéditeur - Spedition Transport-ID

DSV ROAD NV/SA

Agent

Correspondent - correspondant - Ansprechpartner Tel.

Kristel

Plant - fabriek - usine - Werk

SUBS CSM BE04 Olen

Destination - bestemming - Lieferanschrift

1017875

VALE EUROPE LIMITED

Clydach Refinery

Ynyspenllwch Road

SWANSEA SA6 5QR

Client - klant - Kunde

CIT03A202

TODINI AND CO. SPA

CERAMICS & CHEMICALS

Corso Milano 46 Scala B

I-20900 MONZA (MB)

Unloading point - reference - vessel - closing - destination - agent

Losplaats - referentie - schip - sluiting - bestemming - agent

Point de décharge - référence - bateau/embarcation - destination - age

Abladestelle - Verweis - Schiff - Schliessung - Bestimmung - Agent

F201 UK Customs Agent Name: DSV-e

mail: sophie.turner@uk.dsv.com

EORI NR : GB778655171000/delivery to be booked

in advanced

Contact person : Mr. Phil BOWDEN,

phil.bowden@vale.com Tel +44 1792

Shipping marks **2202067826**

Country

United Kingdom

Mat.nr. N° Article	Customs Tariff code - douanetariefcode - Code du tarif douanier - Zolttarife Code Description - beschrijving - désignation - Bezeichnung	Quantity Hoeveelheid Quantité Menge	Net weight Nettogewicht Poids net Netto Gewicht (kg)	Tarra Tara Tara (kg)	Gross weight Bruto gewicht Poids brut Bruto Gewicht (kg)
29405135	2833 2400 Nickel Sulphate Electroless Grade, 22,3%, 1000 kg big bags UN 3077 MILIEUGEVAARLIJKE VASTE STOP, N.E.G. (Nikkelsulfaat Hexahydraat), 9, III, (-) UN 3077 MATIÈRE DANGEREUSE DU POINT DE VUE DE L'ENVIRONNEMENT, SOLIDE, N.S.A. (Sulfate de Nickel Hexahydrate), 9, III, (-) UN 3077 ENVIRONMENTALLY HAZARDOUS SUBSTANCE, SOLID, N.O.S. (Nickel Sulphate Hexahydrate) 9, III, MARINE POLLUTANT Segregation Group: Not Applicable EMS n° F-A/S-F	22	22,000.000	66.000	22,066.000
	Palet - Palette:	22		440.000	440.000
	Totals - totaal - total - Gesamt		22,000.000	506.000	22,506.000

Incoterms: DAP, Swansea

Comments - bemerkingen - commentaires - Bemerkungen

For receipt - voor ontvangst
pour réception - für Empfang

transporter - vervoerder
transporteur - Fahrer

Cobalt Energy Products Olen
Umicore, SA/NV

Watertorenstraat 33 - B 2250 Olen - Belgium
Rue du Marais - 31 - Broekstraat
Tel (32-2) 227.76.36 - Fax (32-2) 227.79.30

Tel (32-14) 24.54.78 Fax (32-14) 24.57.56
B-1000 Bruxelles - Brussel
RCB/HRB 85382 - TVA/BTW BE0401574852

FATTURA / INVOICE

SEDE OPERATIVA E COMMERCIALE / COMMERCIAL OFFICE TODINI AND CO S.P.A. Corso Milano, 46 - Scala B Tel.: ++39 039 230.24.95 r.a. 20900 MONZA (MB) - ITALY Fax: ++39 039 230.24.97 Web: www.todini.com				INTERESTAZIONE FATTURA / INVOICE TO VALE EUROPE LIMITED SWANSEA SA6 SQR CLYDACH GREAT BRITAIN			
P.IVA / VAT NO. IT 10722620159							
DESTINATARIO / END USER FTV C 1900				INVIO CORRISPONDENZA / MAILING ADDRESS VALE EUROPE LIMITED SWANSEA SA6 SQR CLYDACH GREAT BRITAIN			
FATTURA NUMERO / INVOICE NO. 131670		DEL 06/12/23		PAGINA / PAGE 1 / 1		PORTO / DELIVERY TERMS (Incoterms 2020 Apply) DAP	
VS. P.IVA / YOUR VAT NO. GB 778655171		CODICE FISCALE / FISCAL CODE 778655171		VALUTA / CURRENCY USD		CONDIZIONI DI PAGAMENTO / PAYMENT CONDITIONS 45 DAYS INVOICE DATE	
BANCA / BANK INTESA SANPAOLO (*) SWIFT CODE: BCITITMM - IBAN: IT55L0306920407161009352019				BANCA CLIENTE / CUSTOMER BANK			

DESCRIZIONE MATERIALE DESCRIPTION OF THE GOODS	U.M. U.M.	QUANTITA' QUANTITY	PREZZO UNIT. UNIT PRICE	TOTALE AMOUNT	C.L. VAT GROSS
Our DIRETTO TODINI-UK Nr. 00000000083 Date 05/12/2023					
Our ORDER UK Nr. 00000000204 Date 14/12/2022					
Your ORDER UK Nr. 00000000204 Date 14/12/2022					
NICKEL SULPHATE ELECTROLESS	KG	5.000	4,32	21.600,00	207
NICKEL SULPHATE ELECTROLESS	KG	17.000	4,32	81.940,00	207
Origin of the goods: Belgium					
Ref. Loading list nr 0226742 dated 18/12/2023					
TASSA DOGANALE CUSTOMS TARIFF CODE 28332400	NICKEL SULPHATE ELECTROLESS			PER LO CARICO GROSS WEIGHT 22.066,00	PER LO NETTO NET WEIGHT 22.000,00

IMPOSTA DI BOLLO VIRTUALE ASSOLTA AI SENSI DEL DECRETO MINISTERIALE 17/08/2014
CONTRIBUTO CONAI ASSOLTO OVE DOVUTO

The Exporter of the products covered by this document, EXPORTER REFERENCE NUMBER ITREXIT10722620159, declares that, except where otherwise clearly indicated, these products are of EU preferential origin.

Numero EORI / EORI Number Importatore / Importer GB778655171000

CAMBIO / EXCHANGE RATE: 1,0817				TOT. IMPORTABILE / BASE AMOUNT 98.030,88 EUR		TOT. IVA / VAT AMOUNT 0,00 EUR	
PAGAMENTO / DUE DATES 20/01/2024 USD 106.040,00 207		IMPORTABILE / BASE AMOUNT 106.040,00		ESCLUSO IVA ART. 7 bis		TOT. IMPORTABILE / BASE AMOUNT 106.040,00	
TOTALE FATTURA / INVOICE AMOUNT USD						106.040,00	

MULTI-MODAL DANGEROUS GOODS FORM

This form may be used as a dangerous goods declaration as it meets the requirements of SOLAS 74, chapter VII, regulation 4; MARPOL 73/78, Annex III, regulation 4 and IMDG Code

1 Shipper/Consignor/Sender Umicore, SA/NV Industrieweg 34 2280 Grobbendonk BE		2 Transport document number 8728712	
		3 Page 1 of pages 1	4 Shipper's reference 22000000
		5 Freight Forwarder's reference	
6 Consignee VALE EUROPE LIMITED Clydach Refinery Ynyspenllwch Road SWANSEA SA6 5QR UNITED KINGDOM		7 Carrier (to be completed by the carrier)	
8 This shipment is within the limitations prescribed for: (Delete non-applicable) PASSENGER AND CARGO AIRCRAFT CARGO AIRCRAFT ONLY		SHIPPER'S DECLARATION I hereby declare that the contents of this consignment are fully and accurately described below by the proper shipping name, and are classified, packaged, marked and labelled/placarded and are in all respect in proper condition for transport according to the applicable international and national governmental regulations.	
10 Vessel/flight no. and date		9 Additional handling information 24 hours emergency number: Carechem24/Umicore Region Europe: +32 3 213 15 70	
11 Port/place of loading Grobbendonk/BE/			
12 Port/place of discharge		13 Destination Swansea/GB	
14 Shipping marks * Number and kind of packages; description of goods Gross mass (kg) Net mass (kg) Cube (m³) 22 BIG BAG 1000 KG 22.066,000 22.000,000 Nickel Sulphate Electroless Grade, 22,3%, 1000 kg big bags UN 3077 MILIEUGEVAARLIJKE VASTE STOF, N.E.G. (Nikkelsulfaat Hexahydraat), 9, III, (-) UN 3077 MATIERE DANGEREUSE DU POINT DE VUE DE L'ENVIRONNEMENT, SOLIDE N.S.A. (Sulfate de Nickel Hexahydrate), 9, III, (-) UN 3077 ENVIRONMENTALLY HAZARDOUS SUBSTANCE, SOLID, N.O.S. (Nickel Sulphate Hexahydrate) 9, III, MARINE POLLUTANT Segregation Group: Not Applicable EMS n° F-A/S-F SDS : 300000002037 22 Palets 440,000 Total weight 22.000,000			
15 Container identification No./ vehicle registration No.	16 Seal number(s)	17 Container/vehicle size & type ☐ Open ☐ Closed size:	18 Tare mass (kg) 19 Total gross (including tare) (kg)
CONTAINER/VEHICLE PACKING CERTIFICATE I hereby declare that the goods described above have been packed/ loaded into the container/vehicle identified above in accordance with the applicable provisions.** MUST BE COMPLETED AND SIGNED FOR ALL CONTAINER/VEHICLE LOADS BY PERSON RESPONSIBLE FOR PACKING/LOADING.		21 RECEIVING ORGANIZATION RECEIPT Received the above number of packages/containers/trailers in apparent good order and condition, unless stated hereon; RECEIVING ORGANIZATION REMARKS:	
20 Name of company Umicore, SA/NV		Haulier's name BATIM	22 Name of company (OF SHIPPER PREPARING THIS NOTE) Umicore, SA/NV
Name/status of declarant Kimberly Willemsen		Vehicle reg. no.	Name/status of declarant Kimberly Willemsen
Place and date Grobbendonk/BE/		Signature and date 19.11.2013	Place and date Grobbendonk/BE/
Signature of declarant		DRIVER'S SIGNATURE	Signature of declarant
* DANGEROUS GOODS: You must specify: UN No., proper shipping name, hazard class, Packing groups (where assigned), MARINE POLLUTANT and observe the mandatory requirements under the applicable national and international governmental regulations. For the purposes of the IMDG Code see 5.4.1.1. ** For the purposes of the IMDG Code see 5.4.2.			
See overleaf for documentary aspects.			

3

Exemplaire pour le transporteur • Exemplaar voor vervoerder • Exemplar für Frachtführer

LETRE DE VOITURE • VRACHTBRIEF • FRACHTBRIEF (CMR) B N° FEB 210955184

Co transport est soumis, nonobstant toute clause contraire, à la Convention CMR.
Dit vervoer is, ongeacht enig tegenstrijdig boding, onderworpen aan het CMR-Verdrag.
Trotz gegenteiliger Abmachung unterliegt diese Befrachtung den Bestimmungen des CMR-Übereinkommens.

1 Expéditeur ou commissionnaire (nom, adresse, Etat) • Afzender of commissaris (naam, adres, Staat)

Umicore, SA/NV
Industrieweg 34
2280 Grobbendonk

2 Destinataire (nom, adresse, Etat) • Geadresseerde (naam, adres, Staat)

VALE EUROPE LIMITED
Clydach Refinery
Ynyspenllwch Road
SA6 5QR Swansea

3 Prise en charge de la marchandise (lieu, Etat, date, heure) • Inontvangstneming van de goederen (plaats, Staat, datum, uur) • Warenafname (Ort, Staat, Datum, Uhr)

Grobbendonk

4 Livraison (lieu, Etat, date, heure) • Aflevering (plaats, Staat, datum, uur) • Lieferung (Ort, Staat, Datum, Uhr)

Swansea

8 Frais affranchi au transport • Transportgebonden kosten • Transportgebundene Kosten:

- ☐ Temps d'attente • Wachtijden • Wartezeiten
☐

5 Transporteur principal (nom, adresse, Etat, n° de licence, plaque(s) d'immatriculation) • Hoofdvervoerder (naam, adres, Staat, vergoeding, plaatnummer(s)) • Hauptfrachtführer (Name, Anschrift, Staat, Genehmigungsnr., amtliche(s) Kennzeichen)

DSV ROAD NV/SA
Transportcentrum L.A.R.
8930 Rekken

KN-1504G
DSVA-3803421

6 Transporteur sous-traitant (nom, adresse, Etat, n° de licence, plaque(s) d'immatriculation) • Ondervervoerder (naam, adres, Staat, vergoeding, plaatnummer(s)) • Unterfrachtführer (Name, Anschrift, Staat, Genehmigungsnr., amtliche(s) Kennzeichen)

BATIM

Spółka z o.o. sp. K.
33-340 Stary Sącz
ul. Prymasa Stefana Wyszyńskiego 9A
NIP PL 734 100 91 29

7 Transporteur successeur (nom, adresse, Etat, n° de licence, plaque(s) d'immatriculation) • Opvolgende vervoerder (naam, adres, Staat, vergoeding, plaatnummer(s)) • Nachfolgender Frachtführer (Name, Anschrift, Staat, Genehmigungsnr., amtliche(s) Kennzeichen)

9 Réserves du transporteur lors de la prise en charge de la marchandise • Voorbehoud van de vervoerder bij de ontvangst van de goederen • Voorbehoud des Frachtführers bei der Warenabnahme

Przyjęcie towaru przez kierowcę nie oznacza, że kierowca jest odpowiedzialny za ewentualne braki ilościowe towaru, jego sposób opakowania, oraz prawidłowe zabezpieczenie ładunku. Drivers are not allowed to be present at the loading therefore, they cannot be held responsible for any shortages in the amount of goods, their packaging and correct way of securing the goods.

BATIM podpis

10 Marchandises transportées (nature, nombre, poids brut ou net, emballage, marques et n°s) • Vervoerde goederen (naam, aantal, bruto- of nettogewicht, verpakking, merken en nrs.) • Beförderte Güter (Art, Anzahl, Brutto- oder Nettogewicht, Verpackung, Kennzeichen und Nrs.)

NICKEL SULPHATE EN GRADE, BIG BAGS 1000KG 22.000,000KG 22.066,000 KG
SDS : 300000002057 22,000 Pieces

UN 3077 MILIEUGEVAARLIJKE VASTE STOF, N.E.G. (Nikkelsulfaat Hexahydraat 9, III, (-))

UN 3077 MATIÈRE DANGEREUSE DU POINT DE VUE DE L'ENVIRONNEMENT, SOLIDE, N.S.A. (Sulfate de Nickel Hexahydrate), 9, III, (-)

22 Palets
Total weight 440,000
22.506,000 KG

11 Documents annexes transmis par l'expéditeur • Door de afzender bezorgde aanvullende documenten • Documents handed over by the sender

Carechem24/Umicore
Region Europe: +32 3 213 15 70

13 Instructions de l'expéditeur • Instructies van de afzender • Anweisungen des Absenders

- ☐ Contre remboursement • Onder rembours • gegen Nachnahme
☐ Reprise pakets vicks • Terugnans logs praliten • Zurücknahme der letzten Ladepaketen
☐

Delivery number: 8228742
Ref. number : 2202067826
Ref. Customer : OFK 58 (1)

12 Lieu et date d'établissement • Plaats en datum van opmaak • Aufstellungsort und Datum

2280 Grobbendonk 14.12.2023

14

Umicore, SA/NV
Industrieweg 34
2280 Grobbendonk

Kimberly Willemsen

Signature et cachet de l'expéditeur ou du commissionnaire
Handtekening en stempel van de afzender of commissaris
Unterschrift und Stempel des Absenders oder Speditour

15

BATIM
Spółka z o.o. sp. K.
33-340 Stary Sącz
ul. Prymasa Stefana Wyszyńskiego 9A
NIP PL 734 100 91 29

Signature et cachet du transporteur effectif
Handtekening en stempel van de werkelijke vervoerder
Unterschrift und Stempel des tatsächlichen Frachtführers

16

Signature, date et cachet du destinataire
Handtekening, datum en stempel van de geadresseerde
Unterschrift, Datum und Stempel des Empfängers

• Modak verboden op grond van het M.B. van 2015/2014 • Reproducties verboden op grond van het M.B. van 2015/2014 • Nachdruck verboten gemäß ministerieller Erlasse vom 23.05.2014

E-mail: info@ferret.be
http://www.ferret.be

02 425 00 00
02 425 00 66



EUROPESE GEMEENSCHAP

UITVOERGELEIDEDOCUMENT	Afzender/Exporteur (2) : UM RUE DU MARAIS 31 1000 BRUXELLES BE		Nr. RE0401574852		SOORT AANGIFTE (1) EU A indic.ASO (S22)		MRN 23BEE0000078161946		
	Geadresseerde (8) VALE EUROPE LIMITED YHYSPEITWICH ROAD SA650R SWANSEA GB		Nr. geen		Formulieren (3) 1 1 S		Aangewijze (S00) S		
	Aangeven/Verlegenoediger (14) GERLACH & CO SCHOUWKENSTRAAT 7 2030 ANTWERPEN		Nr. BE0404527216		Artikelen (5) 1		Totaal Culti (6) 22		
	Identiteit van het vervoermiddel bij vertrek (18) DSVA3803421				Retentie nummer (7) GB-942022-8228742-2202067826		Datum van afgifte : 19/12/2023 Dowangkantoor : BEHS216000		
	Vervoerswijze aan de grens (25) 3		Plaats van de goederen (30) BEGD5HP00404004		Code betelingswijze vervoerskosten (S29) ---		Code L. verz./uitv. (15) a 33		
Cod. en omschrijving van de goederen (31)	Kantoor van uitgang (26) BE343000				Code L. bestemming (17) s GB		Code(s) land(en) die deel uitmaken van het vervoertraject (S13) ---		
	Merken en nummers - Container(s) nr(s) - Aantal en soort 22 PX 29405135				Verzeglingsnummer (S25) 22506,000				
Aantal (32) Aantal en soort colli, aantal stuks, merken en nummers van de colli (31/1)		Omschrijving van de goederen (S12)		Geadresseerde (8)		Goederencode (33)		Summiere aangifte/Verzeglend document (40)	
Identiteit van het vervoermiddel bij vertrek (15)		Uitvoerennummer van de zending (7)		Nummer(s) van de container(s) (31/3)		Verzeglingsnummer (S25)			
Verzeglende stukken/certificaten (44/1)		Bijzondere vermeldingen (44/2)		Regeling (37)		Land van uitv. (15a)		L. bestamm. (17a)	
UNDG (44/4) 1 22 PX 29405135		Code betelingswijze vervoerskosten (S29)		Scheep aangifte (1)		Scheepische waarde (46)		Nuttomassa (Kg) (38)	
DSVA3803421		28332400 00 0000 0000		NIKKEL SULFATEN					
GB-942022-8228742-2202067826		2325 8228742-2202067826, 2024 BE AROF DBODP45 GDA, 4006 BSLAGTH06404, 4007							
RE0401574852, Y323 ..X040 RE0401574852									
ALGEN07 BE0404527216		10 00		BS		GB		22506,000	
						64728,38		22000,000	

F CONTROLE DOOR HET KANTOOR VAN VERZEGELING/UITVOER

Uitslag:

Aangebachte verzegeling: Aantal:
merken:

Tentoon (uitlaste datum):

CONTROLE DOOR HET KANTOOR VAN UITGANG (K)

Datum van aankomst:

Controle van de verzegeling:

Opmerkingen:

VALE EUROPE LTD
CLYDACH
SWANSEA
SA6 5QR

GOODS IN TICKET

TRANSACTION No 52870

REGISTRATION

DG69ZYZ

SUPPLIER

UMICORE

PRODUCT

NICKEL SULPHATE BAGS

HAULIER

DSV TRANSPORT

DESTINATION

VALE SWANSEA

ORDER No.

21/12/23

CONTAINER No.

DSVA3803421

NOTES

HAT/GLASSES

YES

FIRST WEIGHT

37920 kg

038994

21/12/2023 12:11:5

SECOND WEIGHT

15320 kg

038997

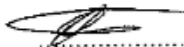
21/12/2023 13:48:4

NET WEIGHT

22600 kg

DRIVERS NAME DAVID

Operators Signature

Drivers Signature 

A record of the weights used on this transaction will be kept for six months

TONDINI UMICORE SULPHATE

22 x 1 TONNE BAGS

BAY 7 POWDER WAREHOUSE

LOT N° - 2940502620

DEL N° - 8228742

BAG DATE - 09/2

Appendix G

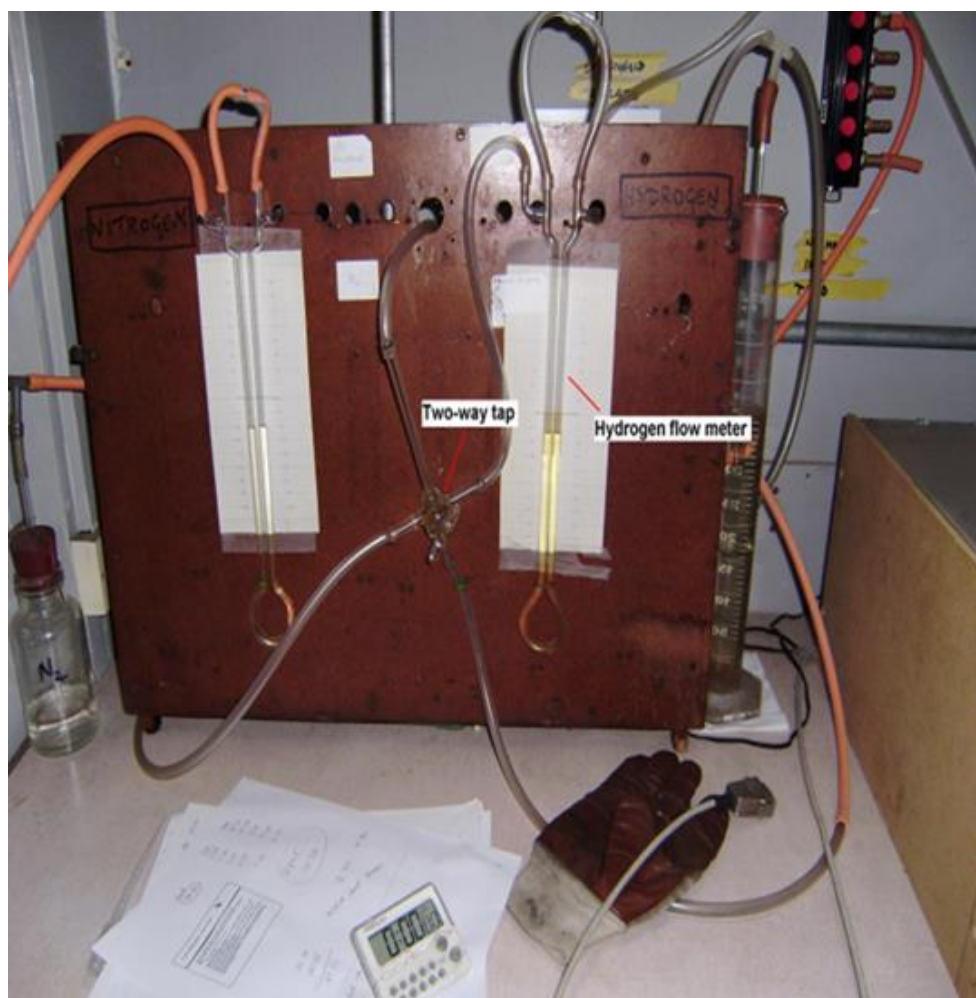


Figure 23, A photograph of the apparatus described in our further work to establish the effects of pH on the Hydrogen Reduction process