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OPERATION AND PROCESS CONTROL DEVELOPMENT FOR A PILOT-SCALE LEACHING AND SOLVENT EXTRACTION CIRCUIT RECOVERING RARE EARTH ELEMENTS FROM COAL-BASED SOURCES

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THESIS

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A thesis submitted in partial fulfillment of the
requirement for the degree of Master of Science in Mining Engineering
in the College of Engineering
at the University of Kentucky

By

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Lexington, Kentucky

Director: Dr. Joshua Werner, Assistant Professor of Mining Engineering

Lexington, Kentucky

2019

ABSTRACT OF THESIS

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The US Department of Energy in 2010 has identified several rare earth elements as critical materials to enable clean technologies. As part of ongoing research in REEs (rare earth elements) recovery from coal sources, the University of Kentucky has designed, developed and is demonstrating a ¼ ton/hour pilot-scale processing plant to produce high-grade REEs from coal sources. Due to the need to control critical variables (e.g. pH, tank level, etc.), process control is required. To ensure adequate process control, a study was conducted on leaching and solvent extraction control to evaluate the potential of achieving low-cost REE recovery in addition to developing a process control PLC system. The overall operational design and utilization of Six Sigma methodologies is discussed. Further, the application of the controls design, both procedural and electronic for the control of process variables such as pH is discussed. Variations in output parameters were quantified as a function of time. Data trends show that the mean process variable was maintained within prescribed limits. Future work for the utilization of data analysis and integration for data-based decision-making will be discussed.

KEYWORDS: Rare earth elements, Process Control, programmable logic controller, Leaching, PID

Douglas Kweku Addo

06/20/2019

Date

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DEDICATION

This work is dedicated to my wife Anita Nti-Addo for her encouragement and patience and everlasting love, my father, and all my family members for their support, love and trust.

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CHAPTER 1. GENERAL INTRODUCTION

1.1 Introduction and Background

The global demand for mineral and metal commodities in an ever-changing world has increased rapidly therefore, geology, mining, chemical and other engineering disciplines are tasked with the responsibility of exploring new mineral and metal sources, developing safe mining methods, determining productive mineral processing technologies, and performing efficient hydrometallurgical extractions and low-cost chemical refining processes.

Maintaining and suitable supply of minerals such as rare earth elements (REEs) for the global need is not just simply a mining, industrial and manufacturing issue, but also a national security issue. In the Department of Energy 2011 Critical Material Strategy report (Bauer et al., 2011), extraction, processing, mineral transformation and user application encompasses the entire industrial supply sequence of REEs utilization. Observing these supply chain steps in Figure 1.1, one sees a dynamic complex combination of both physical and chemical processes to achieve meaningful results (Chu, 2011).

Figure 1.1. Rare Earth Element supply chain (Bauer et al., 2011).

The immediate conclusion drawn in achieving relevant results in rare earth element recovery is the economics and technology. As part of their stewardship, Department of Energy (DOE) has evaluated various rare earth elements critical to the ongoing development and utilization of clean energy technologies. These elements are Yttrium (Y), Europium (Eu), Terbium (Tb), Neodymium (Nd), and Dysprosium (Dy) (Bauer et al., 2011). Due to changes in green energy technologies and shifting markets on a global

scale, the future of the world's energy market is uncertain. To protect domestic critical materials supply in the United States, the U.S Department of Energy has funded numerous research initiatives in the area of alternate REE supply, recovery, and extraction, in the United States.

Although researchers have reported the occurrence of REEs in coal and coal byproducts, literature of recovery processes is limited for coal-based REEs. Zhang et al., 2015 reported that the composition and complex distribution of REEs in coal and coal byproducts, as well as, the non-existent or lab scale REE recovery process for coal products is the reason for such limited literature. To compensate, researchers have been exploring the existing REEs enrichment processes and recovery techniques used on other REEs sources. Typical REE extraction and recovery methods include; roasting and leaching; ion exchange, precipitation, adsorption and solvent extraction (SX) (Honaker et.al., 2018; Xie et al., 2014; Zhang et al., 2018). The successful recovery of metals through acid leaching and solvent extraction has been noted in literature. Research has also presented evidence on the unique extraction and recovery of REEs and other metals (e.g. copper, zinc) through acid leaching and solvent extraction (Zhang et al,. 2015; Honaker et al., 2018).

Regardless of the exact REEs recovery process, the flowsheet will involve a series of complex steps; hence, the development of effective process control is vital to the operation and recovery of critical rare earth elements. The implementation of process control in chemical and mineral-metallurgical recovery processes is critical to the safety, operation, performance, productivity, quality, and overall product recovery (Bascur, 2019).

1.2   Project Objectives

The overall objective of this project was to design and develop a safe, efficient, and low cost process control system with the capability of effectively producing high-grade rare earth elements from coal-based sources. In so doing, a programmable logic controller (PLC) was built to allow automatic control of processes in pilot plant operation. This work demonstrates the operation and control of critical variables using a PLC. The specific project objectives were:

1. Design and deploy a smooth control system to operate pilot-scale leaching and solvent extraction plant with a capacity of up to 1/4 tons per hour.
2. Build and install a working PLC system at the lowest cost possible.
3. Selection of sensors and components on minimal budget.
4. Design and program a graphical user interface (GUI) that is user friendly and allow for easy use and monitoring.
5. Control and collect data for critical variables.
6. Design appropriate safety interlocks.
7. Debug logic program and all necessary instrumentation.

Prior to the completion of the above-mentioned goals and objectives, several milestones are determined and discussed in the project organization.

1.3 Project Scoping

This thesis will discuss the design and development of a PLC control system with the capability of controlling critical variables and effectively producing high-grade REEs. The pilot-scale leaching and solvent extraction rare earth element recovery plant is designed to operate using both Allen Bradley PLC-based automated control system and manual controls. The overall objective of this project is to design and develop a safe, efficient, and low-cost process control system with the capability of effectively operating a pilot plant producing high-grade rare earth elements from coal-based sources based on six sigma methodologies.

Project scoping involved the design of a process flowsheet for the pilot plant hydrometallurgical operation (Figure 1.2). The process flowsheet shows a leaching system, wastewater treatment system, filtration system and solvent extraction circuit. The following chapters will explain the functionality of relevant processes.

By the utilization of Six Sigma methodologies by the research team, a process control plan was initially developed for the leaching circuit system (see Appendix 1). This control plan serves as the basis of maintaining, assessing and documenting the functional steps utilized to guarantee the quality control standards for leaching process. The control plan consist of two main functions, the first function assesses activities performed in

maintaining and improving product and data quality from process, whiles the second function evaluates the accuracy and precision of process.

Furthermore, a safety failure modes and effects analysis (FMEA) which evaluates the plant processes to identify potential areas of failures and relative impacts was created in order to identify and quantify safety concerns and effect change where needed (see Appendix 2).
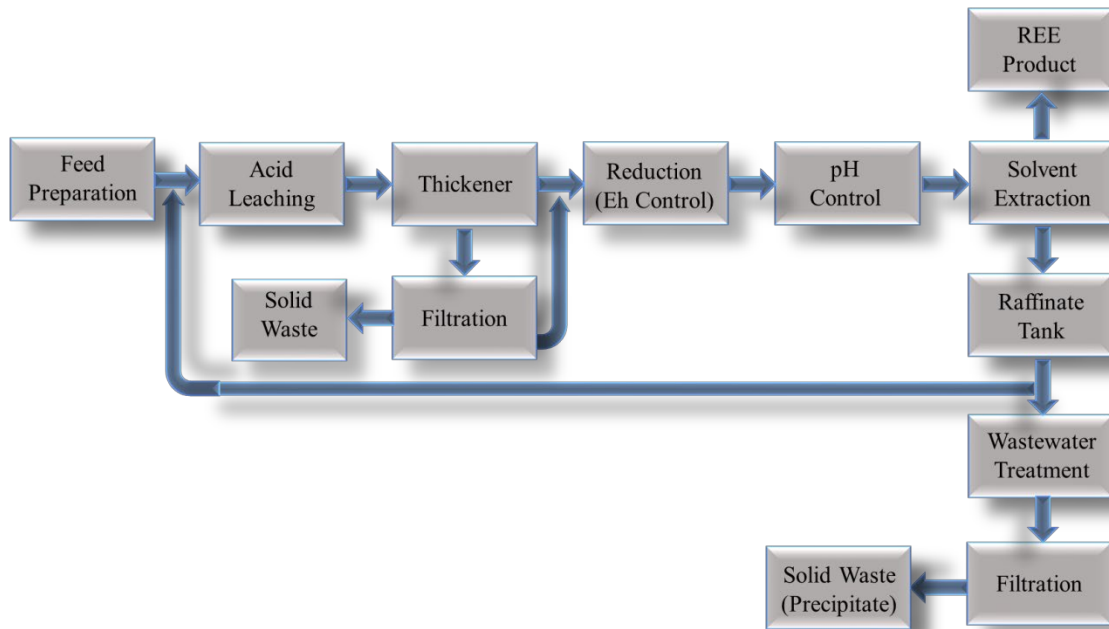
Figure 1.2. Schematic flow diagram of pilot plant process.

CHAPTER 2. LITERATURE REVIEW

2.1   Introduction

Critical to any research project is the characterization of previous research conducted in the field. As such, this chapter serves as a comprehensive review of relevant work completed in areas of chemical process control systems.

The important transition of United States energy sector to a clean energy economy and a leader in the production of clean energy technologies has led the U.S Department of Energy (DOE) to examine the importance of rare earth elements (Bauer et al., 2011). With growing worldwide demand due to emerging technologies and uncertainty in the long-term supply of rare earth elements (REEs), many researchers have been exploring sources and recovery technologies for rare earth elements. Due to the nature of chemical and mineralogical techniques used in the recovery of REEs, the implementation of process control systems in the chemical and mineral-metallurgical processes is critical to safety, operation, performance, productivity, quality and overall product recovery (Bascur, 2019).

Understanding process control systems and how they are implemented in the chemical and metallurgical industry is the crucial step in designing better controls. The literature review outlines previous studies completed on the subject of automation, and mineral-metallurgical process control methods, corresponding to the research project objective.

2.2   Process Control

Over the years, process control has become an integral component of all mineral processing and hydrometallurgical plants. The working definition of process control according to the literature review is largely influenced by one's specific interest. Ormrod et al., 1976 stated that classical process control in the metallurgical industry involved wet-chemical analysis, sampling, and manual adjustment of different process variables. Process control in its broad definition can be interpreted differently for various disciplines, however, for current mineral and metallurgical engineering applications, it is mainly concerned with the maintenance of desired process variables such as pressure, pH,

composition, temperature, flows, concentration and others at some desirable value in a chemical or physical system (Marlin, 1995).

Since the integration of process control in industrial mineral processes, many operations have seen increased performance, productivity, and reduction in the cost of operations. According to literature, advances in computer programming, new instrumentations, cloud technologies, wireless communication and management systems have greatly influenced the growth of process control in the metallurgical processing industry. Similarly, factors such as control actions, parameter measurements, and control strategies that form overall plant control management system also impact process control development in the mineral-metallurgical processing industry (Bascur, 2019).

2.2.1   Why Process Control

Many research works have comprehensively reviewed process control and reported the reason why we need controls in different perspectives. Marlin, 1995 and Stephanopouls 1984 listed the main reasons for process control as safety, environmental protection, plant performance, product quality, operational improvement, and profitability. Achieving the above-listed requirements involves the intelligent use of equipment and sensors, continuous monitoring by the operator, and the quick response to operational variations. Factors that affect the rare earth elements leaching and solvent extraction recovery process include temperature, pH, Eh, percent solids, and residence time (Zhang et al., 2018; Yang et al., 2018).

In the effort to maintaining the desired variables, special attention must also be given to other system input variables termed 'disturbances' existing in the process (Seborg et al.,2010; Bascur, 2019). Although the classical approach to process control in the metallurgical industry is generally dependable, awareness of an operator to changes in the process and taking appropriate action in a timely fashion was cumbersome hence resulting in excessive consumption of reagents (Ormrod et al., 1976). Thwaites, 2007 in Figure 2.1 illustrated that, for any desired process variable, the overall process control goal is to measure and understand initial variability, stabilize and later optimize process constraint.
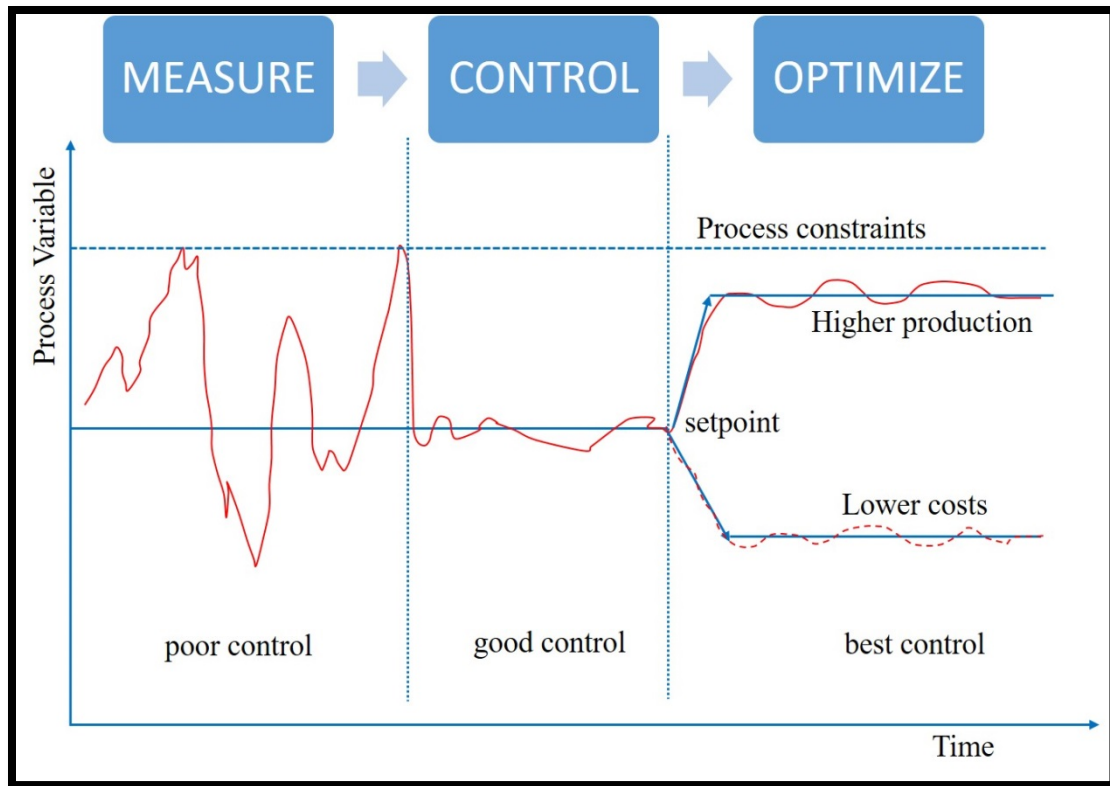
Figure 2.1. Generalized process control goal (reproduced from Thwaites, 2007).

Over the past decade, various industrial processes incorporating process control systems summarizes control objectives into five main general categories:

- safety and environmental protection.
- equipment protection.
- product quality.
- production and profit optimization.
- monitoring and performance diagnosis.

These five major categories of control objectives are discussed in the following subsections (Marlin, 1995; Stephanopouls, 1984).

Safety and Environmental Protection - Safety in relation to process control does not only cover people and environment but also machines used during any operation. Almost every operational activity involving people involves some considerable amount of risk; therefore, the objective of the controls engineer is to implement safety features, practices

and equipment to limit the risk factors involved in each task. Although the current REEs recovery pilot plant operating under certain temperature and atmospheric pressure conditions is designed to operate safely, an incorrect response by an operator to key variables (pH, flow, etc.) in the process can lead to system failure and potential release of harmful chemicals into the environment. To prevent against such failures, automation of critical factors (flow circulation, pumping, valves, and rate of change) ensure desired outcomes.

In a leaching process, the safety control system is purposed to prevent against dangerous and damaging conditions like; drive amperage, power draw, sump level rate change and circulating load (Bascur, 1990). Another critical control strategy to improve human and environmental safety is to include emergency systems (such as E-Stops, or automatic interlocks), which can act as a quick response to electrical faults, equipment malfunctioning, stop spills, shutting down pump systems or even entire plant operation when necessary. REE's metallurgical recovery plants tend to produce large amounts of physical waste (i.e. liquid, solid and gas) that gets discharged into the environment within permit limits. A well strategizes control system implementing recent waste management technologies like pH balancing, recycling, autoclaving and incineration, have the potential to convert hazardous components into useful materials that can be further used in the process (Philip et al., 2009).

Equipment Protection - Every piece of equipment used in any mineral-metallurgical recovery process is specifically designed to operate at some maximum capacity beyond which it will fail. The purchase and repair cost of most of the equipment used in the plant is expensive, hence vital to protect. Literature work also associates increased plant operational cost to equipment replacement and operational delays therefore, it is crucial that each equipment operates within the allowable limits. Similar, the control approaches that ensures the safety of working personnel and environment is applicable when it comes to equipment protection. Implementing emergency control systems to START/STOP operations when problems occur will ensure the maintenance of equipment. Since most pieces of equipment used in metallurgical process are located in environments where they are exposed to harsh temperature and pressure conditions, and high levels of corrosive

chemicals and gases, it is critical to take steps that will protect equipment and increase operational life.

Product Quality - Every chemical or hydrometallurgical plant has product quality specifications determined by the customer. In mineral processing, final product quality may be expressed in terms of percent compositions (e.g., 98% REEs or 20% solids), physical property (e.g., weight, density), or combinations of both. Process control provides real value to plant operation by maintaining the operating conditions needed for product quality. Improving product process control is an economic factor in the application of digital computers and automation control algorithms. The inadequacy of product quality sensors to measure elemental composition or particle size distribution of the dynamic process stream is why process models derived from laboratory scale experiments and samples are used as standards to estimate the quality variables in the process.

Operation consistency and Economic optimization - A metallurgical-mineral processing plant involves a complex matrix of interacting processes, thus requiring consistent smooth process operation that reduces system disturbances. Naturally, the goal of every plant operation is to make a profit. Based on each country of operation, tt is worth noting that before any process control operation can be economical, selected independent variables must be manipulated to meet the first three control objectives (i.e. safety and environmental protection, equipment protection, and product quality). By reducing changes in key variables (pH, temperature, Eh etc.) through process control, plant performance will improve. When variations in key input variables are limited, the desired value of the controlled variable can be fine-tuned, enabling an increase in final profits. In the REEs recovery pilot plant, for instance, the objective of increasing recovery or profits is achieved by proper coordination of all related plant activities while running at optimal throughput. A profitable process control system can be achieved by implementing a control strategy that will absorb the disturbance that the plant process is subjected as well as estimate the indices and constraints. Therefore, operating conditions must be controlled at maximum yields, minimum operating cost, and losses (Bascur et al., 2003).

Monitoring and Performance Diagnosis - Complex mineral-metallurgical extraction and recovery sites require modernized process control automation and monitoring systems. Most of these plant are equipped with both onsite/offsite control and computing systems the generally provides both monitoring and control features to operators and other trained personnel to manage the safe operation of the plant, calibrate sensors and instruments, logic programming and tuning, monitor sampling data, run test experiments, oversee long-term plant performance, environmental monitoring, maintenance checks and more. In a typical metallurgical plant, several display screens showing real-time trends of control processes are installed in appropriate locations since certain areas of the plant may have multiple measured variables. Due to the inability of plant operators to monitor all process variables, plant control systems are equipped with alarm features like flashing lights, sirens, and emergency stops (E-Stops) to improve monitoring. With the advancement in computing and operational control, minor operational losses have reduced through quality and fault prediction algorithms. Performance data is used to identify poor performance areas and opportunities for process improvement (Marlin, 1995; Bascur, 2019; Bascur, 1990).

2.2.2 Industrial Control Systems and Strategies

The basic objective of process control systems is the real-time updating of data to adjust the process tools over a wide range of conditions. A control system either adjusts process based on variable values or hold values of the controlled variables constant. To achieve the plant operating objectives, identifying the best method to limit the variations of controlled variables and the understanding of plant process dynamics is vital. Over the past decade, the more common mode of operating process control systems has been the electrical/electronic method. The definition of control strategy varies from one source to the next. Fundamentally, the two most common strategies is feedback and feedforward controller (Dunn, 2006; Malaterre et al., 1998).

Feedback control - is a common feature of what is popularly known as a closed-loop system in which the output information from the process is used to calculate the value of the controlled variable to correct the desired operational output by the set point. A simplified feedback control system is shown in Figure 2.2. The control compares the

command set point with the output value, and then adjusts the input signal appropriately to produce new output (for example, a pH neutralization system).
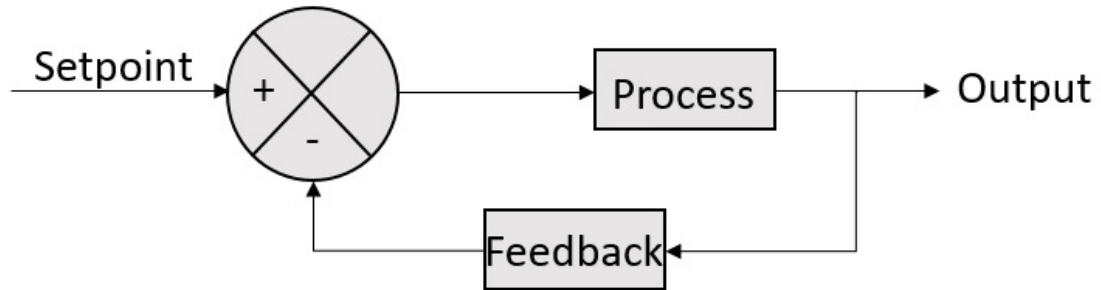
Figure 2.2. Simplified block diagram of the feedback control system.

Feedforward control - on the other hand, does not use the process output to manipulate the value of the controlled variable but rather reduce the effect of input disturbances. The control mode uses the measured input disturbances as an early warning signal to the controlled variable. With sensors to detect process changes or disturbances, the feedforward controller has the opportunity to take calculated corrective action before any feedback control response. A model based advance calculation is needed to implement this control logic, therefore, making this type of control strategy complicated and expensive. Within the last decade, the development of robust control technologies for mineral-metallurgical process applications has increased. Bascur, 2019 outlines four major control strategies needed to optimize mineral-metallurgical processing plants, which are discussed in the following sections.

Expert-based control strategy - In a less complex metallurgical process engineering application, a control strategy can be based on prior knowledge and experience of the plant operator or expert process engineer to determine the control parameters. This type of approach requires some degree of inspection, sampling, and testing to successfully achieve satisfactory plant control. The experienced-based knowledge of the expert is written by means of if-then rules similar to fuzzy logic controller protocols. The rules tend to be the bases for controlling a given system of operation. Some literature reported handling complex control problems with expert rule-based strategy and fuzzy logic controls instead of using mathematical process models (Aydogmus, 2009). Deciding to adopt a model-based control approach or rule-based strategy rest on whether the process disturbances can be modeled with a controller to react accordingly to performances. For many metallurgical processes, the control system will be a combination of the model-and expert based system.

Process Dynamics - Most mineral-metallurgical processes are sophisticated with constant variations of the controlled parameters or outputs over time. Understanding the process dynamics will provide better ways to handle such variations in the process. Dynamic systems change behavior with time as a response to external forces (Aström et al., 2010). Mathematically, certain dynamic process parameters (e.g. temperature, pH and Eh) can be controlled using precise differential equation models and feedback control to ensure material conforms to predetermined standards. Although dynamic process control is widely preferred as a more precise approach for control systems, there are some challenges to its implementation due to intrinsic complexities. For example, all corresponding attributes of controlled variables between the control system, tools and equipment must be known and explicitly quantified (Kent, 2000). The process reaction curve is the best method for identifying reaction times between the controlled and independent variables for a process. Performing time domain modeling using curve fitting and frequency domain modeling on the process reaction curve enables us to obtain a dynamic control model. Design and analysis of a fully dynamic system will involve the separation of the system into a number of interacting subsystems.

Multivariable process control - Currently, advance control strategies tend to achieve a near-optimum condition for each process at a reasonable timespan. Multivariable process control involves a sequential response to different operating control variables whilst taking into consideration other process disturbances to produce the expected product (Malaterre et al., 1998). Thus, the classical single-loop control technique cannot work. For this strategy, a dynamic model is used in control variable calculations. It also calculates the set points of controllers to optimize the control objective function (Mular et al., 2002). Implementation of a model-based control strategy will require unique mathematical models (Smith et al., 1985). By performing the process dynamic test, the model parameters can be obtained. Proper maintenance procedures and tuning of parameters related to the control strategy is very important to reducing input disturbances. With the capabilities of data analytic tools, matching a good model to the current process becomes quite easy through software programming.

Regulatory Control Strategy - Regulatory controls involve the implementation of single input-output control loops to maintain variables at required set point regardless of disturbances (Willis and Finch, 2015). Virtually all regulatory control systems are used with software-based proportional-integral-derivative (PID) controllers as a standard control algorithm (Mular et al., 2002). In mineral-metallurgical plants, the regulatory control system typically includes level, flow rate, concentration, pH, control loops and more. Mineral processing plant control loops are mostly in proportional-integral (PI) mode because the derivative action of the controller is not used.

PID control is the most popular form of feedback control utilized and has been widely covered in almost every control theory literature (Aström et al., 2001). Most control and process engineers are very familiar with this theory and its application; therefore, making the implementation of regulatory control strategies easy. PID control maintains controlled variables close to their desired set point by calculation, therefore, improving overall plant operation. The PID control theory is widely used for the single-loop feedback control system. The regulatory-PID control strategy is universally known such that understanding the uses and limitations is critical to control optimization. Important principles and analysis of the PID controller is discussed in the following sections.

2.2.3 PID Controls (Commonly Used Controller)

A proportional-integral-derivative (PID) controller is by far the most dominating single-loop feedback control algorithm used in industrial processes. Although the simplest method of control is the ON/OFF controller, the PID controller has a long history in most automatic control applications (Dunn, 2006; Aström et al., 2001). Application of the PID algorithm consists of the proper sum of all three-control actions (Visioli, 2006). Normally, continuous industrial process controllers use the proportional action (P) on its own as required. (Jones, 1998). According to Aström et al., 2001, PID control makes up about > 90% of all control loops. In addition, most literature reviewed cite the proportional-integral (PI) component as the most commonly applied algorithm in an industrial process.

The PID control theory is based on error value, $e(t)$ calculated from the difference between the set point, $SP(t)$, and the actual value of a controlled variable, $CV(t)$ by the feedback controller (Novak, 2017; Mitra, 2005; Bascur 2019). The error value $e(t)$, is expressed in following the equation:

$$e(t) = SP(t) - CV(t) \qquad\qquad [1]$$

As defined above, the calculated error $e(t)$, is used to produce a control action (e.g. speed up a pump), which controls the manipulated control variable. The error signal is reduced when the change in the manipulated variable reduces the change in $CV(t)$.

Proportional Action (P) – is the controller response when the output signal is proportional to the deviation in the controlled variable. The proportional control output $C_p(t)$ is directly proportional to the control error signal, $e(t)$, since an increase in error cause an increase to the manipulated variable. The proportional control action is simply represented by the expression:

$$C_p(t) = K_p e(t) = K_p e(t) + U_b(t) = K_p \big(SP(t) - CV(t)\big) + U_b(t) \qquad [2]$$

where, K_p is the controller proportional gain (constant of proportionality), which implements the increase in control output when control error increases. An example of a proportional action would be to control the leach solution feed rate by a pump in a tank to

maintain the level at a desired set point. The value term $U_b(t)$ is a bias term introduced to bring a system to steady state (Bascur, 2019; Visioli, 2006; Mitra, 2005).

Integral control action (I) – is based on the history of the control error occurring in the controlled variable. The integral action reduces the error magnitude to zero by constantly adjusting the manipulated control variable. Mathematically, the integral action is proportional to the integral of the error, according to the expression:

$$C_i(t) = K_i \int_{t_0}^{t} e(t)\, dt, \qquad\qquad\qquad [3]$$

where K_i is the controlled integral gain, needed for time accumulation of error. The integral action gradual adjusts changes in the controlled variable to prevent overshoots (Novak, 2017). In addition, the high gain takes over control of the manipulated variable with long-term load changes to apply control corrections (Dunn, 2006; Visioli, 2006).

Derivative Action (D) – This final control action is based on the rate at which the error is changing. That is to say, while proportional action operates on present error value and integral action is based on past control error values, derivative control action rely on control error values. The derivative control algorithm calculates the rate of change in the controlled variable and applies a correction proportional to the time rate of change error. Ideally, the following can approximate the derivative control mode:

$$C_d(t) = K_d \frac{de(t)}{dt} \approx K_d \frac{e(t) - e(t_0)}{dt}, \qquad\qquad\qquad [4]$$

where the derivative gain, K_d, is the controller output proportional to instantaneous rate. The fast corrective reaction time of the derivative control can cause a large control response even at zero error. Amplitude signals of this controller must be adjusted properly to prevent any potential undershoot or overshoot occurrences. The addition of a low-pass filter for the derivative mode whenever a PID controller is implemented helps limit high-frequency noise. The derivative control is never used alone since it will not respond to a fixed control error value (Bascur,2019; Novak, 2017; Dunn, 2006). Therefore, the combination of the proportional, integral and derivative (PID) control theory can be expressed in terms of the controller output(C).

$$C = K_p e(t) + K_i \int_{0}^{t} e(t)\, dt + K_d \frac{de(t)}{dt} \qquad\qquad\qquad [5]$$

15

The expression above represents the ideal mathematical form for the PID controller. The PID controller utilizing all control modes is shown in Figure 2.3.
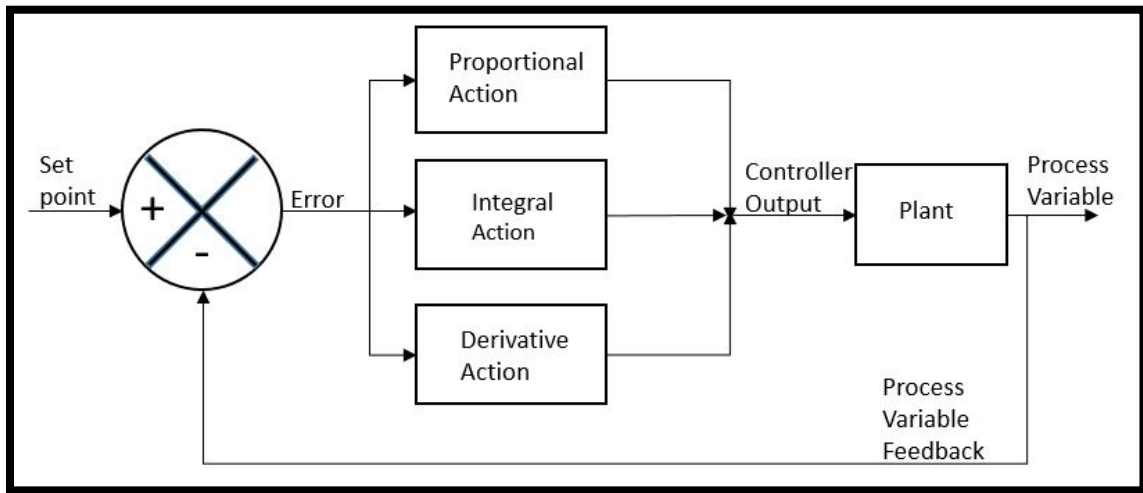
Figure 2.3 Simple block diagram for the feedback control loop PID controller

Dunn, 2006 presented general rules for various types of control loop PID controllers.

- Temperature control application uses PID, with I set for longer time lapse
- Level control application does not require D, but uses P and sometimes I.
- Pressure control process mostly uses P and I actions.
- Flow systems act the same way as pressure controls, using just P and I

In summary, a PID controller utilize three tuning algorithms; proportional gain, integral gain, and rate time or derivative. The use of PID is limited in control processes with high sensor or electrical disturbances (or noise) even after controllers are fine tuned.

Currently, the PID algorithm can be found in many types of stand-alone control equipment in digital forms rather than electrical (analog) or pneumatic components. In addition, the controller can be found as a functional block in programmable logic controllers (PLCs) and distributed control systems (DCS).

Qiao et al., 1996 analyzed the dynamic input-output behavior of a product-sum crisp type fuzzy controller by modeling. The results revealed that this type controller behaves like a parameter time varying proportional-derivative (PD). The research work further

concluded that analysis and designing of a fuzzy control system can utilize conventional PID control theory.

Rathore et al., 2015 performed flow, level and temperature control experimentation using PID controllers along with programmable logic controllers to study the time lapse for heat temperature rise without compromising system stability.

Gao, 2010 established a web-based remote laboratory experiment for control process engineering education based on configuration software and a PLC-based PID control structure. Based on the studies, experimental work became much easier as a result of knowledge of PLC and internet regardless of location restrictions

Chen et al., 2009 designed a fuzzy PID controller that increased the accuracy of a smelting process through PLC-based temperature control method by simulation testing and programming. The results show that this new fuzzy controller performs better than conventional fuzzy PID used in the same application.

2.2.4 Methods of Tuning PID Controllers

Many hydrometallurgical processes extracting and recovering minerals and metals utilize process controllers that apply PID control. Quality of controls can be affected by the accuracy of control devices, control loop stability, and system response to measured variable disturbances. Each section of a plant operation uses a different PID control structure or methodology. The overall setup of the control functions and system design may affect the performance of the process control loop (Dunn, 2006). Tuning of PID controllers in industrial processes simply involves the determination of the control proportional gain value, integral value, and derivative rate value, applied in the right combination to achieve optimum performance.

There have been several methods and theories developed for the tuning of PID controllers detailed in the literature review. Some examples include, Ziegler-Nichols method (Ziegler et al., 1942), Cohen-Coon method (Smith et al., 1985), Tyreus-Luyben method (Luyben et al.,1997), Kappa-tau tuning method (Astrom et al., 1988), internal model controller (Skogestad, 2003; Rivera et al., 1986), Astrom-Hagglund relay method (VanDoren, 2009), backstepping adaptive tuning (Benaskeur et al., 2002), rule-based automatic tuning method (McCormick et al., 1998) and the Nyquist method (Cominos et

al., 2002). These methods can be somewhat simple or difficult to apply due to the use of classical tuning formulae to compute controller parameters. However, the following objectives are met whenever the tuning parameters are well tuned to achieve a stable control system (Ang et al., 2005; Wu et al., 2014; Dunn, 2006).

- robustness against environmental uncertainties
- stability robustness
- robustness against modeling issues
- Noise reduction
- Tracking performance at transient (overshoot, rise and settling time)

Therefore, with the consideration of controller design procedure and varying objectives given, methods of tuning a control loop can be categorized based on their usage and nature as discussed in the following (Ang et al., 2005; Lui et al., 2001; Feng et al., 1999: Kaya and Scheib, 1988; Astrom et al., 1995).

Analytical Methods – The method relies on the plant objective and model to compute the PID controller parameter from algebraic or analytical steps. When applying such methods in hydrometallurgical process control, the control system objective must be in analytical form. An example is the Haalman and λ-tuning method that utilizes a straightforward calculation to determine desired performance from a specified closed-loop transfer function. Similarly, the internal model principle (IMC) with only one user-defined tuning parameter leading to an easy formula that can be used for online tuning (Astrom and Hagglund, 2006; Wu et al., 2014).

Frequency Domain Methods – The application of this method in both industrial and educational settings is more popular than others despite the uncertainties in the plant model. The frequency domain method provides good designs. The optimum PID controller tuning uses the frequency response characteristic of the controlled process to find the parameters required to satisfy system specifications. The only design concern with the frequency domain method is stability robustness.

Trial and Error Methods – These methods require a good understanding of the control loop, engineering skills and practical experience in manual tuning (such as the heuristic Ziegler-Nichols tuning method). These methods do not use an explicit model of the process. If the system is to remain online, these can serve in the form of a rule-based method or in the form of a formula. For these methods, controller tuning is a compromise between operational requirements for stable control and need for fast control (such as the trial and error tuning achieved in the rare earth recovery pilot plant).

Adaptive Tuning Methods – Also termed automatic tuning methods, these utilize one or more of previously established tuning method to obtain controller parameters via auto-tuning. Software programs are available with included features to automatically tune control loops (an example is MATLAB and Rockwell RSLogix 5000), however, these often require fine-tuning for performance optimization.

The basic concepts of control tuning can be found in almost every process control publication or book (Visioli, 2006; Marlin, 1995; Astrom and Hagglund, 1995). An excellent review of tuning methodologies can also be found in (Wu et al., 2014; Liu et al., 2001; Astrom and Hagglund, 2001; Koshkouei et al., 2005). Over the past century, there have been several patented tuning methods. In the United States alone, Table I shows a few patented tuning methods that are mostly rule-based, optimization-based, and formula-based, adopted in industrial for PID design. With all the advances in PID control tuning methods, none replaces the Ziegler and Nichols (Z-N) tuning rule in terms of simplicity, popularity, and ease of use (Ang et al., 2005).

Table 2.1 United States Patents on PID Tuning

Year	Patent Number (US)	Title	Assignee/Owner
1992	5159547	Self-monitoring tuner for the feedback controller	Rockwell International Corporation
1992	5166873	Process control device	Yokogawa Electric Corporation
1993	5223778	Automatic tuning apparatus for PID controllers	Allen-Bradley Company Inc.
1993	5272621	Method and apparatus using fuzzy logic for controlling a process having dead time	Nippon Denki Garasu Kabushiki Kaisha
1994	5331541	PID control unit	Omron Corporation
1994	5335164	Method and apparatus for adaptive control	Universal Dynamics Limited
1996	5568377	Fast automatic tuning of a feedback controller	Johnson Service Company
1997	5691615	The adaptive PI control method	Fanuc Ltd
1998	5818714	Process control system with asymptotic auto-tuning	Rosemount Inc.
2000	6081751	System and method for closed loop auto-tuning of PID controllers	National Instruments Corporation
2001	6253113	Controllers that determine optimal tuning parameters for	Honeywell International Inc.

		use in process control systems and methods of operating the same	
2003	6510351	Modified function blocks in a process control system	Fisher Rosemount Systems Inc.
2004	6697767	Robust process identification and auto-tuning control	The National University of Singapore
2005	6847954	Control-loop auto tuner with nonlinear tuning rules estimators	Fisher Rosemount Systems Inc.

2.3   Implementation of Industrial control systems

Nowadays, there is a wide application of several types of industrial control systems (ICS) and corresponding instrumentations across chemical, mining, and metallurgical-mineral process industries. Industrial process control evolved when physical control mechanisms were replaced by the current system infused with information technology (IT) capabilities. For example, mechanical analog gearboxes replaced by embedded digital controls (Stouffer et al., 2011). An ICS generally consist of a combination of mechanical and electrical components working together towards a process objective. For mineral – metallurgical process control, crucial components of the industrial control system include the control loop, diagnostics and maintenance utilities, and human-machine interface (HMI) as described in Figure 2.4 (Falco et al., 2002). The control loop involves remote sensor evaluating measurement variables, the controller generating command signal after interpreting sensor signal that is used to control elements such as control pump. The HMI displays process information such as system faults and safety alarms revealing any process issues to the operator allowing for any configuration of controlled parameter, set points and control actions. Diagnostics and maintenance tools allow plant operator to monitor and provide quick response to any malfunction or system property changes.
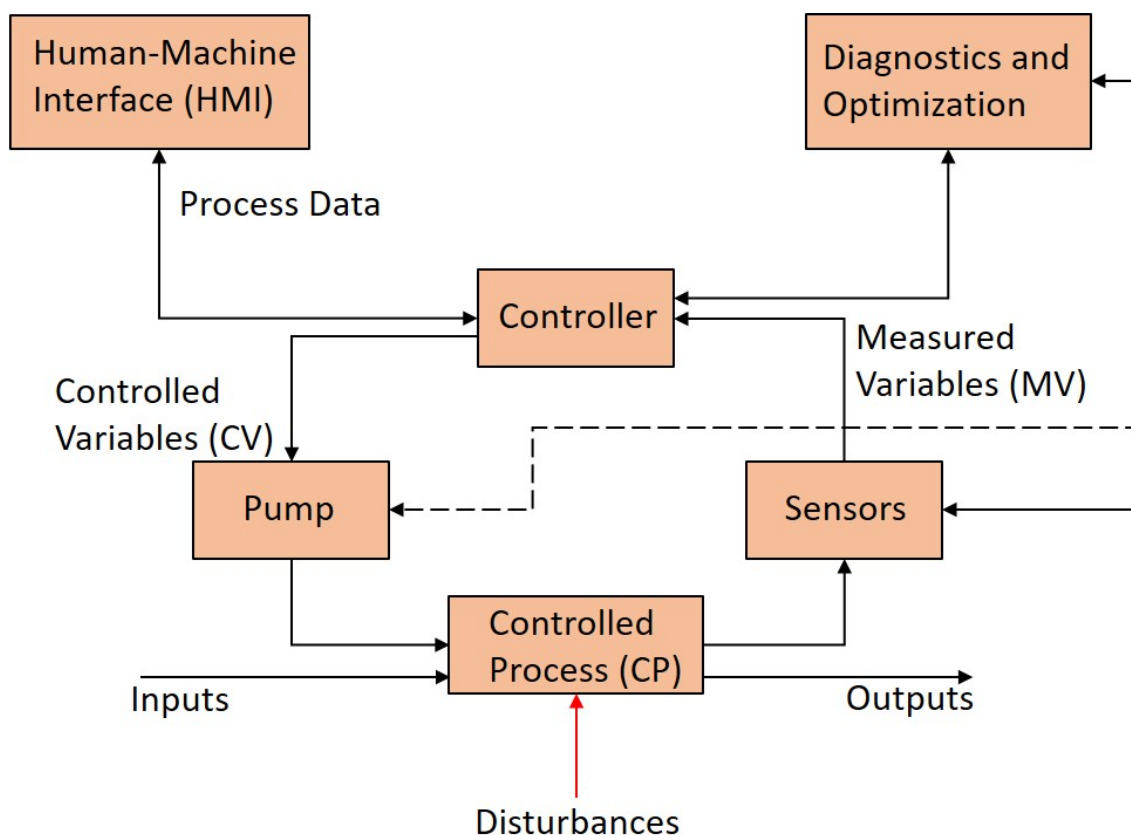
Figure 2.4. Components of industrial control system operation (reproduced from Falco et al., 2002).

A broad set of control systems generally implement an ICS, supervisory control and data acquisition (SCADA)-based system, distributed control systems (DCS), and programmable logic controllers (PLCs). SCADA systems are computer based systems typically used for remote monitoring and control of dispersed process components and analyzing real time data. The DCS are used for large complex system such as chemical mineral refining plants. PLCs are often utilized as control component in both SCADA and DCS systems in present automation processes found in small industrial sectors with few control loops and numerous Industrial PC systems (Ray et al., 2015; Falco et al., 2002). In addition, the SCADA system consists of both software and hardware that generally allows human supervisory controls, acquiring and transmitting data over large geographic regions, whiles DCS provides real-time monitoring information for site-specific control production (Mack, 2018).

Industrial control systems (ICS) can also be either discrete-based or process-based (Rao et al., 2017). The simple discrete-based control system manages discrete sections of single control loop batch chemical processes. A process-based control manages continuous multi-loop process such as wastewater treatment in a metallurgical plant. Currently, most industrial automated controls are restricted to data acquisition by remote terminal units (RTU) or programmable logic controllers (PLCs). The SCADA system analyzes real time data points corresponding to either input or output instrument.

Ray et al., 2015 implemented automation control through SCADA supervision and data logging by the supervisory server at a coal leaching pilot plant. In addition, intelligent control systems embedded in a commercial programmable logic controller for wastewater treatment has been developed (Manesis et al., 1998).

2.3.1 Programmable Logic Controllers (PLC)

Programmable logic controllers or PLC are industrial computer-based, solid-state devices that takes data from instrument sensors and sends commands to actuators to implement controls imitating the behavior of electric ladder diagram as described in the sections above (Alphonsus et al., 2016; Rullan, 1997). The wide use of PLC in mining, chemical and metallurgical process industries is due to their efficiency and reliability in harsh environments. PLCs ability to handle a wide range of inputs and outputs has made them the favorites of automated systems in most industries. For a long time it has been introduced as a robust device that is ubiquitous and provided a unique field of research, development and application, mainly for process industries and college education (He et al., 2015). Figure 2.5 shows a simplified process model of a metallurgical application where a PLC might be used.
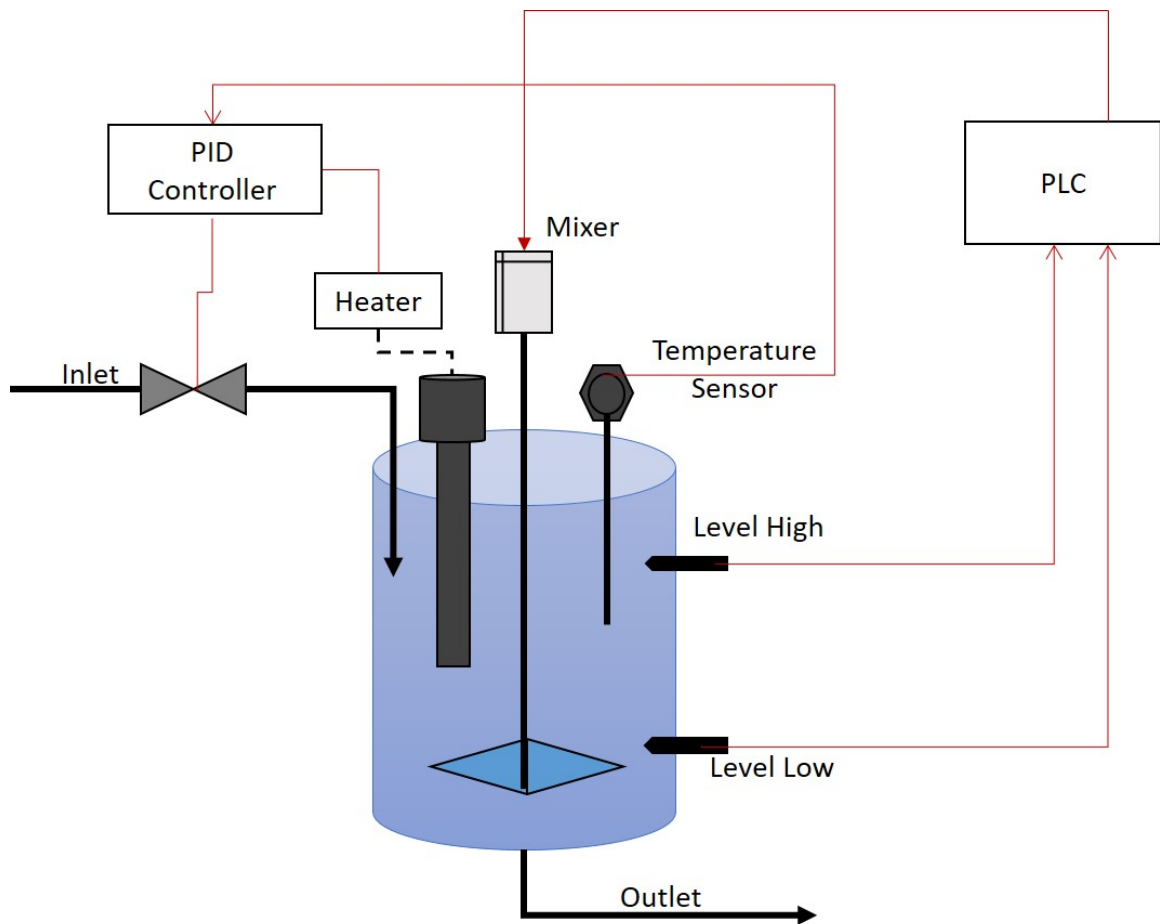
Figure 2.5. Simplified stirred reactor vessel process.

The PLCs have a programmable memory for storing instructions for the purpose of providing specific timing, counting, input-output (I/O) control, sequential and combinatorial logic, arithmetic, communication, data processing, PID control for industrial or non-industrial automation processes (Stouffer et al., 2011; Walker, 2012). PLCs contain two parts i.e. the hardware and software programming (Alphonsus et al., 2016). Like any computer system, the PLC hardware consist of components such as power supply, programming devices, central processing unit (CPU) or controller as well as multiple inputs and outputs that simulate relay and switches to the control device using transistors and other circuitry (Ruban, 2008). Figure 2.6 shows the system components of a PLC presented by Suresh (2015) and Walker (2012).
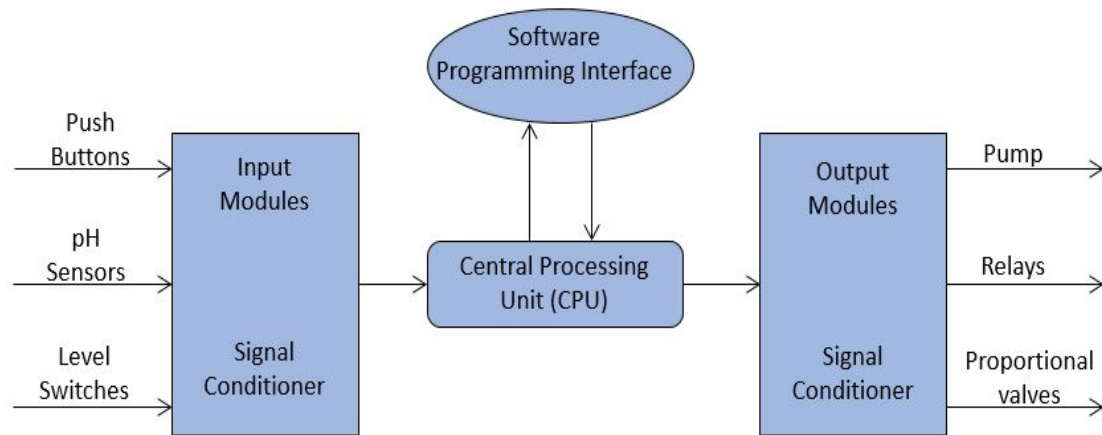
Figure 2.6. PLC system components.

The input module sends conditioned signals (i.e. analog 0-10V or 4-20mA) from sensor and instruments to the controller or CPU component to be processed. The output module relates conditioned signals to operate pumps, relays, proportional valves or other plant equipment under control. The logic term used in the name PLC is due to widely used ladder-logic programming method popularly called the ladder diagram. This ladder logic is a graphical language resembling electrical circuits that uses relay coils and contacts to control external inputs and outputs. Programming is via standard computer interfaces and network options. Figure 2.7 shows an example ladder logic (Bhojasia, 2019) that can be used to control the Figure 2.5 process (Rullán, 1997; Ruban, 2008).

There are other programming languages for PLCs. Listed programming languages in the International Standard IEC 61131-3 for PLCs are function block diagram (FBD) –based on logic-gate symbols; instruction list (IL) – a low level language similar to machine assembly language; structured text (ST) – high-level language similar to a basic computer programming language; sequential function chart (SFC) –another graphical programming method with powerful structuring capabilities not very popular amongst the others (Alphonsus et al., 2016; Tiegelkamp et al., 1995; Frey et al., 2000).

Figure 2.7. PLC ladder logic program for continuous stirred vessel.

There hundreds of companies that supply programmable logic controllers (PLCs) in different sizes and characteristics. The primary companies include Rockwell Automation, ABB Group, Omron, Siemens, and Altus (Ruban, 2008). Details on Allen Bradley Rockwell Automation PLC hardware and programming is discussed in a later section. Innovation in technology to improve industrial and non-industrial process control has seen the high rise of industrial personal computers (PCs) that could be used for PLC purposes.

2.3.2   PLC versus Industrial PC

Industrial PCs (IPC) are said to be able to function effectively for the same industrial process as PLCs (Rullan, 1997). A modern day PLC has evolved to include capabilities of advanced PID control, safety, motion control, and standard PC features, like networking tools and web server. A developed modern PLC is flexible and reliable computer-based device capable of consistent performance in challenging environments that contained high levels of vibrations, contamination, and electromagnetic disturbance. Recently, the industrial PC has successfully infiltrated a number of industrial applications, with accelerated innovation, increased processor speeds and decreasing cost. In spite of the fact that both machines are computers there are a number of factors that differentiate PLCs and industrial PCs (Lipson et al., 2011; Mitra, 2005; Liang et al., 2011).

Some unique similarities between the two technologies relates to the integrated hardware and architecture, both have the capability to control devices using industrial networks,

central processing unit, motherboard, and memory slots with expansion capabilities (Alphonsus et al., 2016). The difference between PLCs and PCs is that PLCs mixes scan-based and event-based (ladder logic) program execution. Industrial PCs in the other hand runs event-based software capable of executing multiple task simultaneously in any order. PLCs are designed with human machine interfaces (HMI) displaying plant process and operational status, whiles industrial PC are furnished with different size monitors (Rullan, 1997). When the intended application of the equipment is to automate a process with little human input or advanced interface, then PLC is the best option compared to industrial PC. Generally, PLCs perform well if the application and equipment operation is simple. An example is a pumping system or wastewater process control (Fernandez et al., 2010; Manesis et al., 1998).

Considering maintenance and troubleshooting capabilities, PLC systems have been designed to make such task easy for plant operator. Troubleshooting of an industrial PC with complex programming codes can be overwhelming if plant operators do not understand the programming language. (Bacidore, 2017; Bystricanova et al., 2011; Aydogmus, 2009). The choice of a PLC system based on cost is dependent on the system specifications, the type of application, and physical environment.

2.3.3   Limitations and Benefits of PLC

Unsurprisingly, the automation of mineral-metallurgical processes in the world today through PLC systems has grown exponentially. In the past, PLCs were commonly used in auto manufacturing applications (Aydogmus, 2009). In metallurgical industries, the complete automation to achieve high productivity and increased profits involves some degree of investment risk. The implementation of PLC-based semi or fully automated systems can help achieve the process goals, but also comes with some benefits and limitations (Alphonsus et al., 2016).

Limitations of PLC

In comparing PLCs to industrial computers for in process control, clearly there are several limitations to the capabilities of PLCs. The use of PLC systems to perform modern complex calculations, advance measurements, networking, and monitoring

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compared to industrial PC has declined tremendously. Listed below are a few limitations of a PLC technology.

- PLC system has fixed circuit operation. Assembly and wiring connections tend to be very tedious and difficult.

- New generation of PLC technology becomes available every three to five years compared to IPC technologies (software and hardware) being updated every few months (Rullan, 1997).

- Regardless of the powerful nature of PLC, response time for process control operation is still not very fast (nanoseconds) (Aylen, 2004).

- PLCs tend to usually have long hold-up time when a problem occur. System error are very difficult to trace, requiring a trained engineer.

- While IPC provides integrated solutions that incorporates the functions of the PLC, programming terminals, and HMI, PLCs do not (Alphonsus et al., 2016).

From the advantages and limitations of PLCs versus industrial PCs, automatic control system has taken a new direction in automation technology. Liang (2011) and Wang (2014) discussed PLC models and implementations of soft PLC running technology based on IPC according to the formulated international standards IEC61131-3. Soft PLC is based on IPC, compared with regular PLC implementation, can meet the modern industrial automation requirements. The studies highlight the soft PLC to have an open architecture, increased data processing capabilities, and strong network communications capabilities.

Benefits of implementing PLC

This section describes several advantages associated with the use of PLC systems. Benefits include systems architecture, procedure, controls, information processing, and implementation. The order of advantages does not reflect any priority in terms of impact or possibility of occurrence. Listed below are some benefits of PLC (Rullan, 1997):

- Robustness – the standard PLC has no moving parts, hence durable in various environments with disturbances (noise), high vibration levels, high temperature, and humidity levels.

- Construction approach – PLCs have modular interfaces already inside the controller allowing for easy additions of components and maintenance (e.g. inputs and outputs (I/O)

- PLCs are easy to understand, easily programmed and reprogrammed with a commonly understood programming language (e.g. ladder logic). Standard computer-based software for programming PLCs allow both online logic rung edits and offline edits and downloads (Alphonsus et al., 2016).

- PLCs have low power consumption, better ability to retain system data after any power failure. A faster restart capability that improves process downtime

- Application-base, programmable logic controllers can be coupled with computers in industries to handle a broad range of analog signals and fuzzy PID loop control programming. (Chen et al., 2009).

- Cost of many PLC systems are relatively cheap compared to industrial PC for the same applications.

Several research studies have shown how PLC applications have improved a system. Example is the automation and control applications in aluminum rolling mills using PLCs (Rao et al., 1995). Another study done by Hartescu et al, 1998 on an integrated system designed for an agglomeration factory (iron ore, coke, limestone and dolomite) and pre-heaters. Process optimization found that a network of PLCs and controlling algorithms implemented increased efficiency and combustible savings. Xianzhong et al, 2009 highlighted the characteristic and functions of a fieldbus technology through adjustment of pH in zinc metallurgical process using Siemens PLCs as the control system.

2.4 Process Control in Metallurgical Industries

Historically, dependable process control in metallurgical industries has involved simple sampling analysis, wet-chemical analysis, and manual control techniques as previously mentioned. The inception of good process control technologies in the metallurgical industry has resulted in reduced time lags between changes in the process and operator responses, improve operational safety and overall profitability, which is lacking in classical process control approach. Management of profitable metallurgical plants rely

primarily on throughput, recovery, and cost with the control system and business data playing a critical part. The PLCs, distributed control systems (DCS), and supervisory control and data acquisition (SCADA) control systems provides real-time data for control of process and display to the operator (Bascur et al., 2003). There are a number of ways by which various process control approaches have benefited the metallurgical industry.

An example is, the implementation of multivariable control scheme for a gold mine milling circuit. The control scheme application resulted in the optimization of the plant operation and reliable control of plant dynamics (Hulbert et al., 1990). Similarly, Van Breusegem et al., 1996 applied a linear quadratic multivariable control to cement milling circuits. The result was an optimized stable separator and mill circuit that produces quality product. Multivariable controller implementation and software programming was done using a regular PC that can be interfaced with other existing control systems or PLCs.

Bascur et al., 2003 presented current information technologies applied to the development of dynamic performance monitoring and integrated workflow systems in metallurgical plants. For process control purposes, the application of real time performance management (RtPM) system in an iron and steel complex enabled the continuous improvement of profits. The system utilized unifying analysis methods to give individuals in an organizations real-time intelligence to continuously improve performance.

Various control systems and strategies have been widely used for many metallurgical applications, for example, motor control, system monitoring, leaching process, extraction research, and more. Some specific process control applications are reviewed in the following subsection.

2.4.1   Leaching process controls

Ray et al. 2015 presented a coal chemical leaching pilot-plant control system that is designed to increase the yields of the coal product. Operation of these leaching plants occur in very harsh environments with dangerous chemicals, vibrations and disturbances that can potentially pose a safety risk to people equipment and environment. The process described employs two levels of automation control. The first level of control is

implemented by a control logic PLC with device net, and control net, as well as intelligent sensor instruments. The second level builds on forward and backwards database functionality between SCADA and server. Control net and standard industrial Ethernet is the system communication medium for sensor and actuators, SCADA and supervisory system connection to PLC controller. PLC function is to send a signal to control feed flow, temperature in pressure vessel, stop vaporization in pressure vessel by air insertion, and control cooling system by proportional-integral (PI) method.

Wu et al. 1999 proposed a model-based expert control system (MECS) to determine and track the optimal pH of the overflows of a continuous zinc leaching process in an extractive circuit. Leaching in zinc hydrometallurgy is an important step to forming zinc sulfate solution after dissolving zinc-bearing materials in dilute sulfuric acid. Effective control is imperative since conventional mathematical model based control methods was not satisfactory. The expert control system is a computer program that combines both steady-state mathematical models and rule models. The main component of the MECS are an expert controller (EC), an automatic measurement system (AMS), and three 761 series single loop controllers connected by voltage converter and wiring relay box as well as pumps and flow meters.

Ye et al., 2017 investigated the real time optimization (RTO) of gold cyanidation leaching process (GCLP) in a two-layer control framework that integrates self-optimizing control (SOC) and modifier adaptation (MA). The study utilizes the lower layer of the control system to track the self-optimizing controlled variables measurements at optimally set points to account for parametric disturbances. The upper layer involves the optimization of SOC controlled variables set points in modified framework to help tackle the plant-model mismatch. Mukae et al., 1976 invented and patented an automatic leaching system for hydrometallurgical production of zinc. The system comprises a feedforward and feedback control circuit used to control the pH of the process slurry at a constant value by controlling the flow rate of the supplied spent electrolyte. Further information on the invention can be read in the reference citation.

Dash et al., 2014 designed and developed a SCADA-based control system for chemical leaching pilot plant treating Indian coal. The strong control system implemented at the pilot plant efficiently regulated plant parameters, improved data acquisition, and

optimized plant operation. To achieve high-level process control of the leaching process, plant automation involves Rockwell-based PLC and other industrial instruments. Overall research findings showed the successful implementation of PLC-based automation and control system helped fulfil the objective of regulating process variables and optimization.

2.4.2   Solvent Extraction process controls

The theory and principle behind solvent extraction (SX) has gained wide acceptance in modern hydrometallurgical industries. With the growing demand for rare earth elements and other metals of high purity, solvent extraction gains importance in hydrometallurgical recovery of metal ores, due to new organic extractants developed for complex leach solution. Over the past years, analytical chemists in laboratories have exploited solvent extraction technique by using organic solvents for the extraction of molecular ions of interest from aqueous solutions. However, the objective for the adaptation of solvent extraction in the metallurgical industry is quite different from the analytical approach (Michaud, 2017; Jha et al., 2002). The solvent extraction process involves two major parts, the extraction step - which applies to the reaction and mixing of two immiscible liquids in a manner to make the transfer of desired ions from one phase to the other feasible; separation in achieved by the selectivity of different ions from the loading and unloading of the organic phase with recyclable organic solvent (Michaud, 2017).

Literature reviewed showed SX process as a unique technique for the recovery and/or purification of zinc, copper, REEs and other compound without the use of PLC-based process control (Kordosky, 2002; Jha et al., 2002; Xie et al., 2014). The solvent extraction metal recovery processes is not a standalone operation but complimentary to the preceding metal leaching circuit and the metal recovery and/or refining process that follows. The properly designed mixer-settler unit of a solvent extraction circuit will result in improve settler throughput (Kordosky, 2002). Application of different process control strategies and PLCs requisite to solvent extraction includes pH control, oxidation-reduction potential (ORP) control, temperature and pressure monitoring, pumping control, recycling rates, and more.

Bergh et al., 2006 describes copper solvent extraction supervisory control system that is designed for studying measurements and control problems in a setup pilot plant. The solvent extraction process is relatively slow with major problems such crud formation, organic entrainment in the aqueous phase, and phase disengagement etc. The innovative control schemes applied are local, metallurgical, and hydrodynamic supervisory controls (Figure 2.8). Furthermore, control architecture incorporates PLC, controllers, and industrial PC networks. The main function of the PLC is to receive input signals from field measurements, communicate output signals, and manages emergency shutdown procedures.
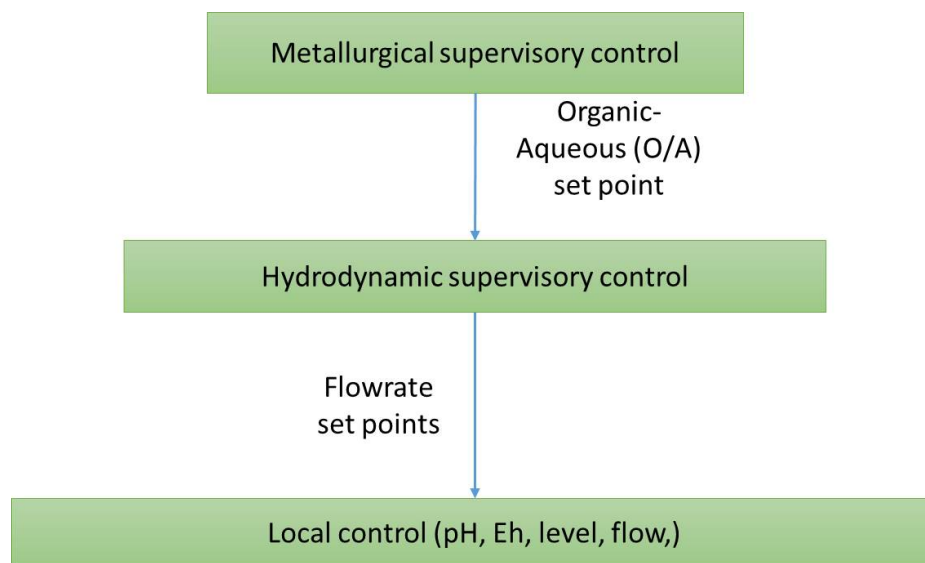
Figure 2.8. SX plant supervisory control architecture (reproduced from Bergh et al., 2006).

Komulainen et al, 2009 studied two level control strategies of an industrial copper solvent extraction process that stabilizes and optimizes copper production by using two stabilizing single-input-single-output controllers, an optimizer and four feedforward PID controllers for regulating process flow rates. Furthermore, process modeling and PI controller is implemented by MATLAB and tuning achieved through internal model control (IMC) rules. Other process instrumentation includes level measurement, temperature, pH and, conductivity measurement in the mixer unit.

Wenli et al. 2000 made a study on the steady and dynamic performance of automatic control system solvent extraction process by using the on-line expert system for rare earth

countercurrent extraction (ESRECE) simulation, and an energy dispersive X-ray fluorescence (EDXRF) analytical technique set up in a laboratory (Figure 2.9). The experimental procedure was to reduce complications with solvent extraction of different rare earth elements and help solve environmental pollution problems. Their findings indicate that the usage of ESRECE simulation software and EDXRF analysis was favorable.
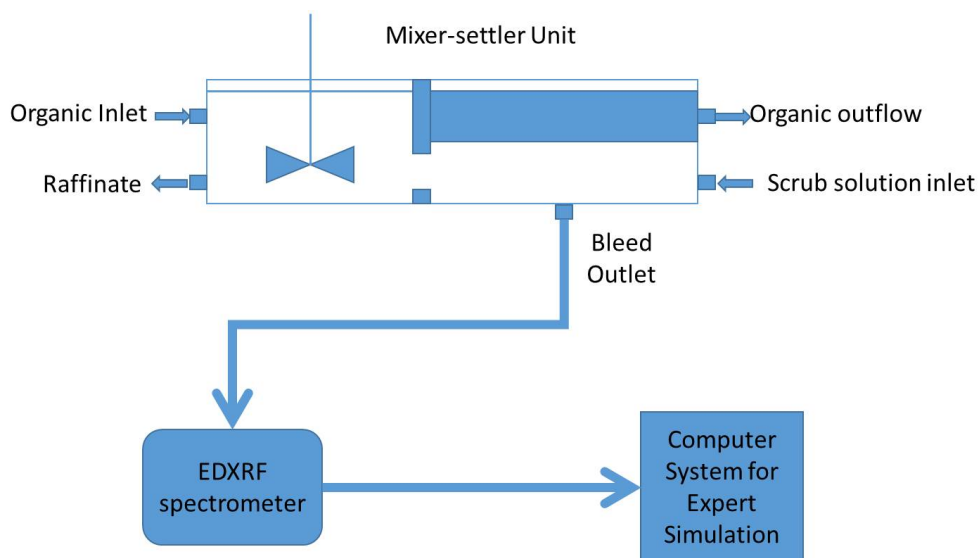
Figure 2.9. SX process automatic control system scheme (reproduced from Wenli et al., 2000).

Anthemidis et al., 2009 developed an automated on-line micro-extraction sequential injection system for metal pre-concentration and separation in water samples. The sequential injection system is equipped with an internal six-port valve, and a 1000µl capacity syringe pump. Control and programming of the control system was done with the aid of a regular windows-based personal computer. The system proved to be reliable and successful in interfacing the continuous operating solvent extraction process with sensitive electro thermal atomic adsorption spectrometry technique and achieved extremely low levels of organic solvent consumption.

2.4.3 Effluent controls

He et al., 2013 describes an automated electrolytic manganese wastewater treatment system designed to meet environmental requirements and resource utilization. The wastewater generated from such manganese industries poses serious environmental pollution concerns. The control systems consist of a PLC, industrial PC, expansion modules, external measurements, and control devices. Control system implementation is by the PLC-based controller, ion exchange control, and recycling treatment control process. The control system operates in two modes, manual operation and automatic mode according to PLC-based PID control schema determined by process complexity.

Ali et al., 2009 made an experimental study to determine the flow rates of a small capacity water pumping system using integrated PLC and frequency inverter. The PLC system determines the related time intervals between the flow rate levels and flow rate set time, considering control of the dynamic temperature change between different operating points by the frequency inverter. The software mathematical model of the water pumping system was developed on MATLAB-SIMULINK. The system proved successful in representing the real behavior PLC and frequency controlled water pumping system.

Baeza et al., 1999 developed a real-time expert supervisory system for the monitoring and control of a wastewater treatment pilot-scale plant based on two autonomous process computers and PLC. The real-time expert system developed in G2 software actuates and controls supervisory set points whilst maintaining plant performance in normal conditions. Furthermore, the PLC controlled pump units, level sensors, nipping valves, pH sensor, temperature sensor, dissolved oxygen sensor, ORP sensor and other mechanical units of the plant. The communication system developed in C language uses Transport Control Protocol/Internet Protocol (TCP/IP) for the maintenance of a real time database in the server.

Wareham et al., 1993 researched the implementation of ORP as process control parameter for automatic control of wastewater treatment systems. Their findings clearly indicate that ORP probes demonstrate promise for automated control of anoxic sequences in wastewater treatment systems.

CHAPTER 3. PILOT-SCALE PLANT DEVELOPMENT

Chapter 3 outlines the critical initial review and research performed in order to achieve control objective, plant operation, in addition to methods and materials used to complete PLC system development for pilot-plant process control. This includes shakedown testing with acid and/or base solutions and instrumentations. Additional information about the type of PLC system and modules used for plant automation is described in this chapter along with specific sensor instruments and equipment used for data collection.

In the following subchapters, the control theory, PLC setup, overall system design, and plant operation is discussed.

3.1   Control Theory

After careful interviews and deliberation with project team members, numerous points were noted which list the select group of variables (percent solids, temperature, pH, ORP, residence time, water control) that are critical to the operation of the plant leaching and solvent extraction circuits. The initial control theory of the plant operation, especially the leaching circuit will take into consideration all of these critical points based on the circuit piping and instrumentation diagram (P&ID) shown in Figure 3.1.

- Percent solids: The percentage concentration is expressed as weight/volume percent or mass/volume percent. The variation measures the amount of solid (solute) in grams and measures amount of solution in liters. The solids feed of 20% solids by volume in Tank 6 (TK-6) is controlled by rate of solids addition. To achieve the correct 20 percent solids needed for leaching, solid and water will be fed into the system at known rate via PLC.

- pH control: Control of pH will be achieved in leaching Tank 6, Tank 4, and Tank 19. From the P&ID flow diagram, there are four (4) potential streams into Tank 6, two (2) potential streams into Tank 4, and three (3) possible streams into Tank 19. For Tank 6, these streams are raffinate from the solvent extraction (SX) circuit, makeup water, acid makeup solution, and direct solids feed or thickener underflow from Tank 2. For Tank 4 the inputs are sodium hydroxide (NaOH)

solution and outflow from reducing Tank 3. Tank 19 inputs include raffinate bleed from Tank 15, effluent from laboratory operations, and NaOH solution. These tanks will be pH controlled to adjust the rate of acid addition to achieve the pH set point in Tanks 4, 6 and 19. To address control system stability for pH, the following measures are proposed for Tank 4 and 6. The pH will be averaged over a reasonable time period to reduce fluctuation (noise) in readings that may potentially affect the PID control. Alternatively, for Tank 6, Tank 2 thickener underflow pump (P-2) will be slaved to the rate of the TK-1 solids feed rate with a time averaged float point in such a way that TK-2 will not have solids build up. Hence, stabilizing the operation of underflow pump (P-2). The Pump (P-15a) will be regulated by time averaging of the Tank 15 level using a level sensor to produce a near uniform output flowrate to Tank 6. The makeup water will be controlled by either using a simple inline flow meter and proportional valve adjusted to a known flow rate or by using a PID controlled peristaltic pump based on level in Tank 13. Thus, the water addition rate will see a near steady state rate due to the constant flow rate of the circuit bleed on pump (P-15b).

- In the reduction Tank 3, an ORP sensor will help convert ferrous to ferric using a reducing agent in a similar manner as in pH neutralization.

- Temperature: The principle control of temperature is vital in leaching Tanks 6 to 10. Industrial heaters with built-in thermocouples installed on Tank 6 to 10 will control the required temperature for the leaching process either manually by operator or by PLC. The addition of acid into Tank 6 will generate additional heat.   Aqueous solutions above 35°C reporting to the SX circuit can be detrimental, causing excessive evaporation of the organic. A thermocouple installed in neutralization Tank 4 may be useful in monitor the temperature going into SX circuit. Cooling prior to SX might be required to prevent excessive solvent loss. This can be achieved by the addition of simple, inexpensive heat exchangers or increasing residence time.

- Residence Time: The solids residence time control is primarily determined by the flow rate of pump (P-13). The water in thickener Tank 2 is separated into two streams, gravity overflow and underflow by pump (P-2). Excess water will report

to the SX circuit and later reporting to Tank 6 via raffinate holding Tank 15. Pump (P-2) will control the liquid recirculation load in the leaching circuit.

- Water control: The amount of fresh leach solution (raffinate) introduced into the system is controlled by pump (P-15b). This is the effluent pump feeding the wastewater treatment circuit Tank 19. The amount of water utilized in the plant operations is added into Tank 6 by time averaged level control in Tank 13 or by constant flow rate feed. Two streams of water feeds Tank 13, gravity overflow from the leach circuit thickener from Tank 12 and the pulsed or unsteady output water flow generated from the filter press (FP-1). Liquid level in Tank 13 will experience fluctuations over the course of two hours, which is the approximate cycle time of the filter press system. The makeup water control into Tank 6 will need a longer moving time average window to maintain a near steady state water addition to the tank.

3.1.1 Assumptions

The continuous plant operation is bound to have variability in the different process circuitry based on the formulated control theory. In the design of the plant control system, the following critical assumptions are made to achieve operational objectives.

- Variability of Feed Material: The feed rate of solid coal material is closely monitored to maintain the required percent solids feeding the leaching circuit Tank 1 or Tank 6. The system will be designed to deliver between 1 and 20 percent solids to accommodate any changes in flowsheet designs. Reduction in percent solids will occur from Tank 6 to Tank 10 due to acid leaching and water dilution.

- Filter Cake: The slurry feeding the plant or filter cake generated from the filtration will be composed of approximately 25% water and 75% solids.

- Drift in pH: Throughout the circuit, pH is anticipated to drift from Tank 6 to Tank 1. The variability of the coal (ore) will dictate the final pH value in Tank 1 in conjunction with the pH set point value in Tank 6. From the initial process flowsheet design, feed rate, and recirculation rates will be set constant and the acid additions will vary according to the pH set point.

3.1.2 Piping and Instrumentation Diagrams (P&ID)

The complex chemical steps involved with the safe operation of the rare earth recovery processes at the pilot plant usually require mapping out all mechanical and chemical steps with piping and instrumentation diagrams (P&ID). The P&ID or detailed process diagram represents the technical realization of the whole plant process in details through graphical symbols of piping, equipment, measurements and control functions. In the event of a failure in the operational process, reviewing the P&ID is usually a good start point to compare the current state to the ideal state. The importance of P&ID is vast when used in the mineral-metallurgical or chemical industry. For the pilot plant processes these includes:

- Provides information to evaluate process construction
- Serves as the basis for automation control programming
- Develop safety guidelines, operation standards for the pilot plant
- Documentation to explain how process works
- Provide cost estimate recommendations for process.

Project implementation phase also consists of process flowsheet design and P&ID design for the pilot plant processes. Figure 3.1 and Figure 3.2 shows the P&ID of the pilot plant leaching circuit and wastewater treatment circuit respectively.

Figure 3.1. Overall pilot plant leaching circuit P&ID (unscaled).

TO PLC SYSTEM

From TK-15 Page 2 Solution Bleed to Effluent Treatment

To waste Liquid holding Tank

P-21

TK-19

V-19

Lime
Ca(OH)2 Slurry
Make-up Tank

TK-20

V-20

3 cu.ft
Filter Press
(80 gal to fill)

FP-2

P-20

LEGEND

Float Valve (FV)	Level Control	Control Cable to PLC
Flow Indicator	pH Controller	ORP Controller
VFD -SEW	Pump	
Filter Press	Drain Valve	
Level Sensor (LS)	Three- Way Valve	
Immersion Heater	PLC SYSTEM	

THIS DOCUMENT CONTAINS INFORMATION PROPRIETORY AND CONFIDENTIAL TO MINING ENGINEERING DEPARTMENT, UK

TITLE: Wastewater Treatment Circuit
University of Kentucky

CONTRACTOR:

PROJECT: ¼ TON REEs LEACHING PROCESS | SHEET 2 OF 2

DRAWN: DOUGLAS ADDO | DWG No. | REV

DATE: 04-01-19 | 00 | 00

SCALE: N.T.S

Figure 3.2. Plant wastewater treatment circuit P&ID (unscaled).

3.1.3 Safety Interlocks

The safety of the pilot plant operation is of paramount priority. Operation of these process circuits can present safety concerns. As part of process controls, strategically implementing interlocks and alarms will help prevent process failures. The designed interlocks used in the pilot plant is as follows.

- The pilot plant operation will have a master ON/OFF switch to start/stop operation during emergencies. In the event of power failure, the operator using the standard operating proceeding (SOP) must restart the plant. The plant will not automatically resume operation without manual intervention.
- Emergency stops (E-Stops) are strategically located on power system boxes, HMI screen, and other VFD control boxes at strategic locations throughout the plant. Example in Figure 3.3.
- If the liquid level is below the leach tanks heater low level sensors, agitators for the leach tanks and holding and/or surge tank, and pumps will stop.
- If the liquid level within the leaching tank is above the heaters low level sensors, agitators must start.
- Alarms are placed throughout the plant. Overflow alarms and messages are used on Tank 13 and 15 and other aqueous storage tanks in the plant circuit.
- Controls strategy gives priority to emergency switches during the occurrence of any incident.
- Start temperature heaters only if tank liquid level is above the heater low level probe and close all openings to prevent heat loss.
- Before starting pumps, recirculating and discharge valves must be open. Pump tubing is inspected for leaks before operating pumps.

Figure 3.3. Agitator control box with emergency stop button (E-Stop).

3.1.4   Startup and Shutdown

Startup and shut down should have both manual and automatic provisions. For startup of pH control, solid feed/ slurry addition, water addition, level and temperatures should be controlled and monitored to ramp to operational set points. In shutdown, the prevention of line packing by slurry settling, particularly in leach Tanks 1, 2, 6 to 10 and pumps P-2, P-10 and P-14 will be of paramount importance. Agitators must remain operational even after hours to prevent solids settling and clogging in the leach circuit and to maintain a uniform mixture of aqueous and organic solution in solvent extraction circuit. The startup and shutdown procedures for the different process circuits is discussed further in later subsections.

3.2 Setup of the PLC Control Box

A PLC system was determind to be the best option for the pilot plant control system against an industrial PC. Several brands of PLCs on the market were considered, but Allen Bradley (AB) 1769-L33ER CompactLogix from Rockwell automation was the most appropriate for the pilot plant operation. The Allen Bradley PLC system was chosen because of the low-cost price option due to the partnership between the Mining Engineering Department of the University of Kentucky and Rockwell. In addition, working knowledge of Allen Bradley ControlLogix acquired through automation systems course offered by the Mining Department was a factor in selection. The AB 1769-L33ER CompactLogix 5370 L3 controller present an integrated RS-232 serial port, dual EtherNet/IP, 2MB memory, 32 Ethernet IP nodes including 1769 SDN communication interface module for remote device configuration over DeviceNet. Controller comes with a 1GB SD card and integrates with 1769 compact I/O modules with 16 I/O expansion and provide seamless bridging with data collection and control over same network. Control is achieved through computer-based software, RSLogix 5000 by Rockwell. From the programming software, various I/O channels can be assigned to different instruments.

The Figures 3.4 to 3.7 shows the complete automation assembly of the PLC box and the configuration of the Allen Bradley based PLC system and components. Arrangement and installation of the modules as shown in Figure 4.4 involves inserting the correct modules in their respective location by verifying the type, voltage requirement and defined slot address as documented by the manufacturer. With the help of din rails, electrical components such as the controller, local I/O modules, terminal blocks, power supply, and circuit breakers are mounted in the enclosure box. The single feed terminal blocks are used for wire-to-wire termination or connection between instruments and the PLC as shown in Figure 4.5. As shown in Figure 4.6 the PLC system is powered by a 24VDC power supply mounted at the bottom part of the waterproof enclosure box alongside an ethernet switch and miniature circuit breakers for the 7 inch display screen, ethernet adapter, and controller.

Reasons for the PLC control box setup include ease of troubleshooting during failures, allowing for remote module expansion when needed, ease of visual inspection and

operation during testing, flexibility to connect many machines, reduced wiring and safeguard against electrical failures.

Figure 3.4. Allen Bradley PLC setup configuration with all I/O components.
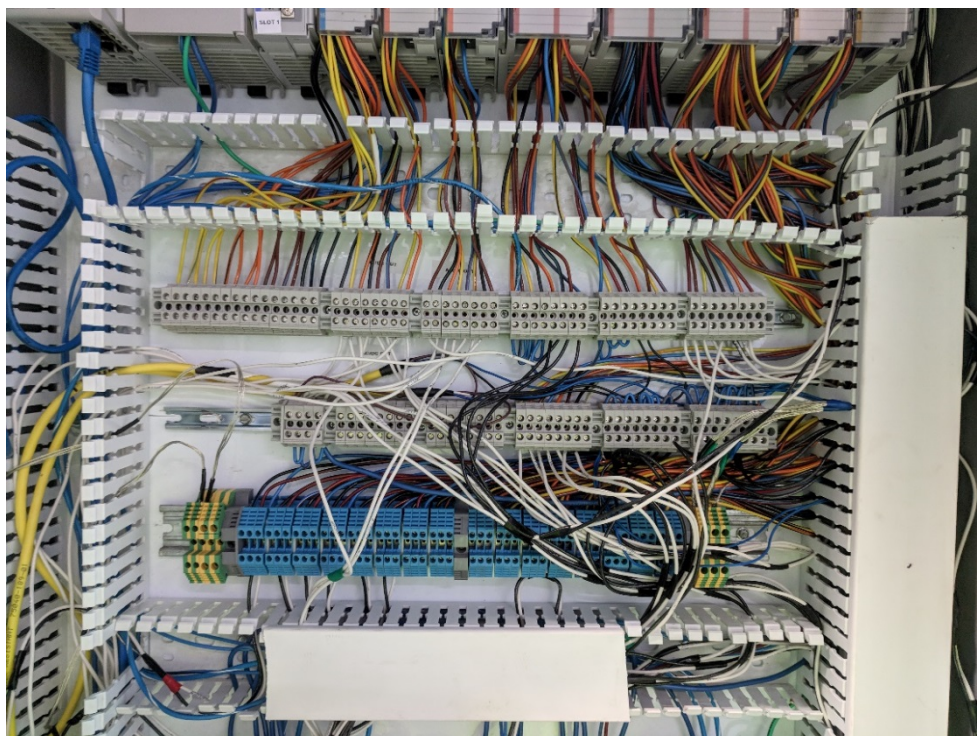
Figure 3.5. Din rail terminal blocks for wire-to-wire connection.
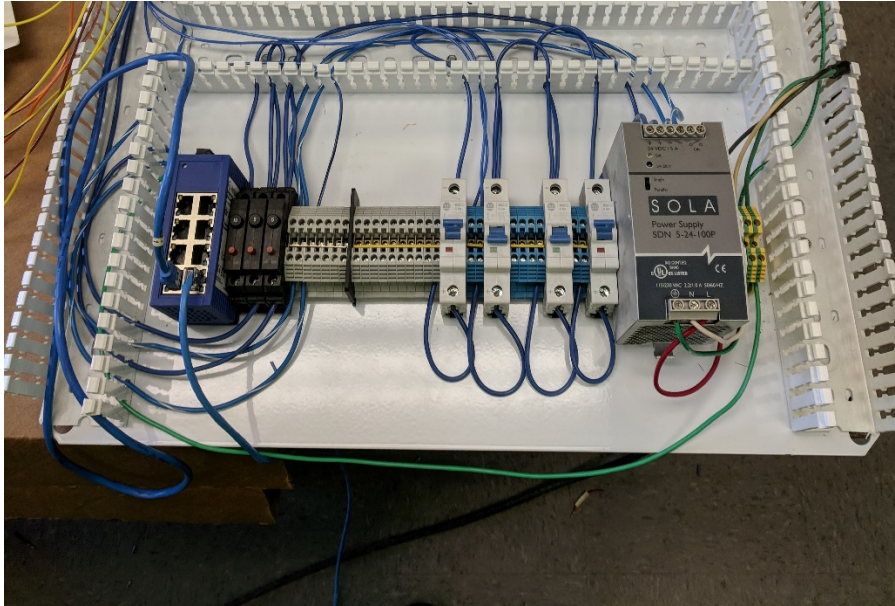
Figure 3.6. PLC box power supply, Ethernet adapter and circuit breakers.
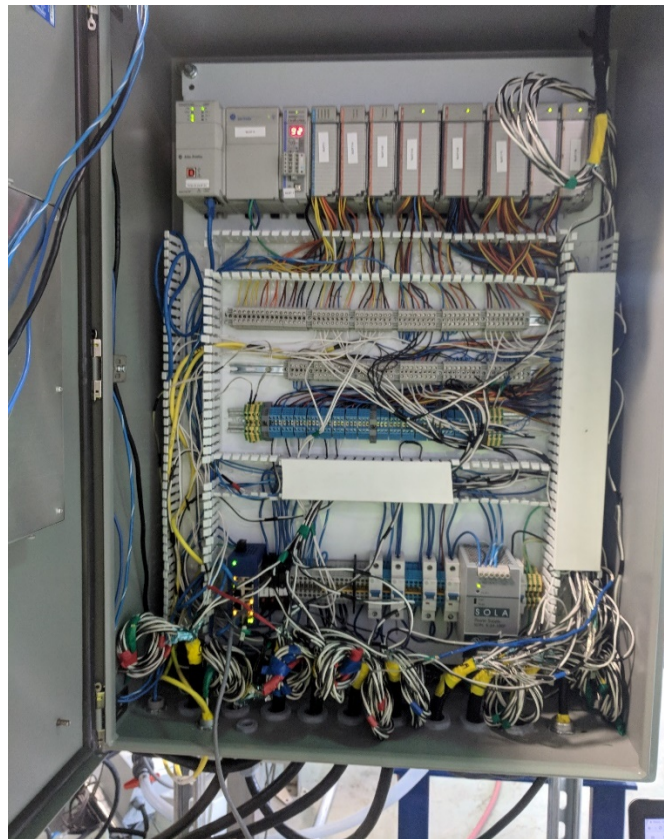
Figure 3.7. Complete PLC box setup.

Furthermore, Table 3.1 list all the various hardware components as assembled in the control box shown in Figure 3.4 above from left to right.

Table 3.1. List of component modules used to build PLC system.

PART/CATELOG NUMBER	MODULE SLOT LOCATION	MODULE DESCRIPTION
AB 1769-L33ER	0	Allen Bradley 1769-L33ER CompactLogix
AB 1769-PB4	-	Allen-Bradley CompactLogix Power Supply 24vdc/4A
AB 1769-SDN	1	Allen-Bradley DeviceNet Scanner Module
AB 1769-IQ16	2	Allen-Bradley CompactLogix 16-Point 24VDC Input Module
AB 1769-OW8	3A	Allen-Bradley CompactLogix 8-Point Voltage Relay Output Module
AB 1769-OW8	4B	Allen-Bradley CompactLogix 8-Point Voltage Relay Output
AB 1769-IF8	5A	Allen Bradley CompactLogix Analog Input Module
AB 1769-IF8	6B	Allen Bradley CompactLogix 8Channel Analog Input Module
AB 1769-IF8	7C	Allen Bradley CompactLogix 8Channel Analog Input Mod
AB 1769-OF8C	8A	Allen Bradley 8 Channel Analog Output Module
AB 1769-OF8C	9B	Allen Bradley 8 Channel Analog Output

3.2.1   Input and Output (I/O) Channels

PLC input channels are used to monitor plant devices such as pH sensors, ultrasonic sensors and other switches. The selection of input module type results from the various types of input devices used at the pilot plant. Some devices respond to either ON/OFF digital inputs whilst others respond to analog signals, which represent process conditions via current or voltage signals. The outputs control devices such as pumps, motors, and proportional valves. Figure 3.8 shows examples of the input and output module component installed in the PLC In operation, the output channel converts signals into either analog (4-20mA) or digital signals. Table 3.2 list all input and output control channels for the complete plant process control. The total number of channels needed for process control influenced the acquisition of the following types of I/O modules. For pump and switch controls, two Allen Bradley 1769-OW8 CompactLogix relay output module with 8 normally open VAC/VDC points is used. Three AB 1769-IF8 analog current 8-point input module are also used for loop powered sensors and other devices, and two AB 1769-OF8C analog current 8 point output module to control corresponding field instruments (example, pumps).
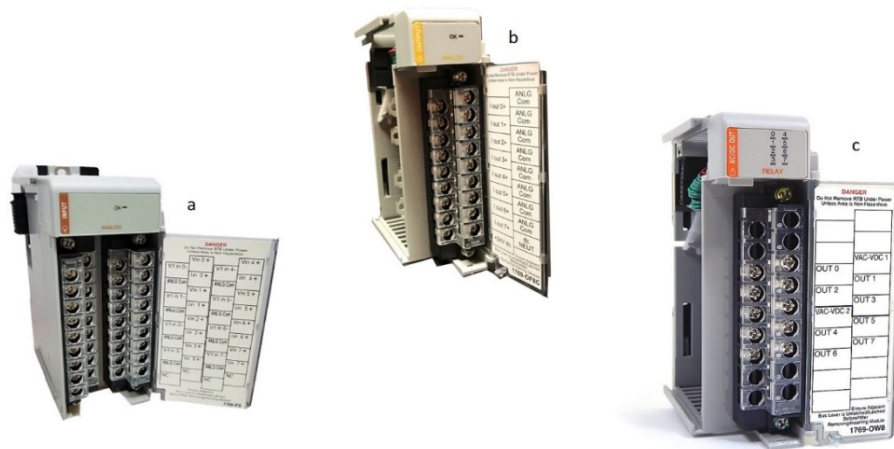
Figure 3.8 Typical components of the PLC, (a) AB 1769-IF8 analog input module (b) Analog output module (1769-OF8) (c) Digital output module (1769-OW8)

Table 3.2. The I/O channels for plant wide process control.

Channel no.	Digital input Dc	Digital output ac/dc	Analog input 4-20ma	Analog output 4-20ma	Description	Use
1			X		Tank 4 pH	For Monitoring & Control
2			X		Tank 6 pH	For Monitoring & Control
3			X		Tank 19 pH	For Monitoring & Control
4			X		Tank 3 ORP	For Monitoring & Control
5			X		Tank 15 Level Sensor	For Monitoring & Control
6			X		Tank 13 ultrasonic sensor	For Monitoring & Control
7			X		Cleaner Unit feed pH	For Monitoring & Control
8			X		I/P Pump 1	For monitoring pump speed
9			X		I/P Pump 2	For monitoring pump RPM
10			X		I/P Pump 3	For monitoring pump speed

11			X		I/P Pump 4	For monitoring pump RPM
12			X		I/P Pump 5	For monitoring pump speed
13			X		I/P Pump 6	For monitoring pump RPM
14		X			L/S Pump 1	For Start/Stop control
15		X			L/S Pump 2	For Start/Stop control
16		X			L/S Pump 3	For Start/Stop control
17		X			L/S Pump 4	For Start/Stop control
18		X			L/S Pump 5	For Start/Stop control
19		X			L/S Pump 6	For Start/Stop control
20		X			L/S Pump 7	For Start/Stop control
21		X			L/S Pump 8	For Start/Stop control
22		X			L/S Pump 9	For Start/Stop control
23		X			L/S Pump 10	For Start/Stop control
24		X			I/P Pump 1	For Start/Stop control
25		X			I/P Pump 2	For Start/Stop

						control
26		X			I/P Pump 3	For Start/Stop control
26		X			I/P Pump 4	For Start/Stop control
27		X			I/P Pump 5	For Start/Stop control
28		X			I/P Pump 6	For Start/Stop control
29				X	I/P Pump 1	For speed control
30				X	I/P Pump 2	speed control
31				X	I/P Pump 3	For speed control
32				X	I/P Pump 4	speed control
33				X	I/P Pump 5	control pump RPM
34				X	I/P Pump 6	output the speed of pump
35				X	L/S Pump 1	For speed control
36				X	L/S Pump 2	speed control
37				X	L/S Pump 3	control pump RPM
38				X	L/S Pump 4	For speed control
39				X	L/S Pump 5	speed control
40				X	L/S Pump 6	control pump RPM
41				X	L/S Pump 7	speed control
42				X	L/S Pump 8	control pump RPM

43				X	L/S Pump 9	For speed control
44				X	L/S Pump 10	speed control

3.2.2 Wiring Diagrams

The correct wiring sequence for connecting plant devices and equipment to the I/O modules is critical to the normal operation of the PLC and other systems. Method of connecting wires to each module is not standardized, hence can be performed at the discretion of the controls engineer but should be performed logically and neatly. The wide distribution of sensor devices and equipment across the pilot plant resulted in a distributed controls wiring setup. Based on the number of I/O channels and location of allocated equipment, smaller size electrical control boxes were built and setup at remote location. This approach made wiring bundles and terminal connections much easier. Figure 3.9 and 3.10 shows junction boxes installed for connecting various distributed process devices to the PLC (for example pumps and pH sensors). Each junction box was setup primarily for pump control relay with pass through instrumentation wire connection. Each box has a circular pin relay switch as well as sixteen (16) terminal blocks. The wiring connection diagrams for the PLC, instrumentations and remote mini enclosure boxes were designed using Microsoft Visio studio as shown in Figure 3.11 to 3.13. In addition, the Table 3.3 and Table 3.4 list the wiring connection sequence from instrument sensors to each channel and wiring pairs for pump cables as shown in figures below. Wiring is checked to have the correct gauge and size to handle the maximum current. The use of wire bundling helped simplify connections to each I/O channel.
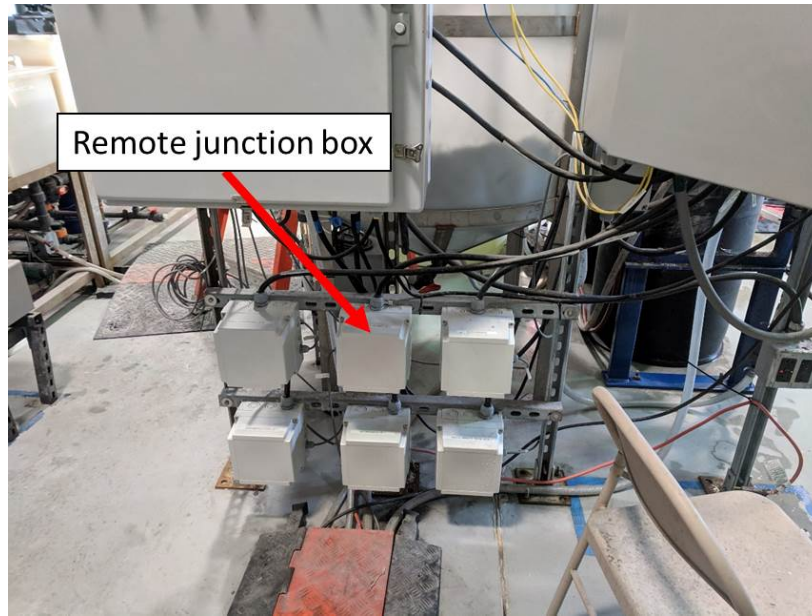
Figure 3.9. Remote junction box located around SX and wastewater circuit.
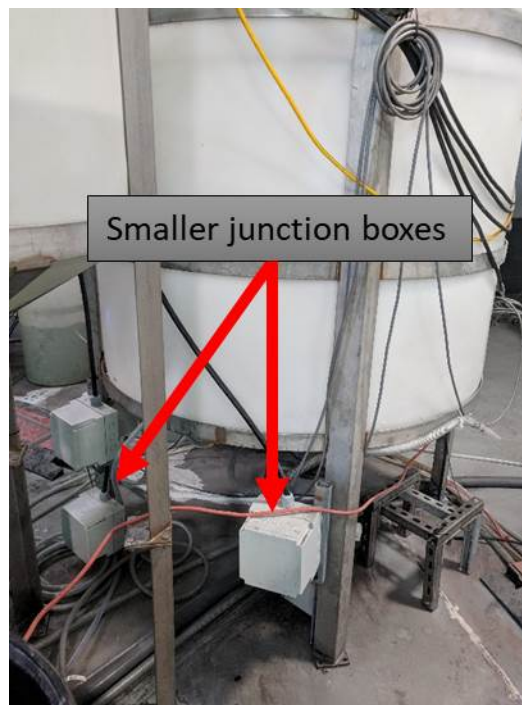
Figure 3.10. Leaching area junction boxes connecting instruments to PLC.
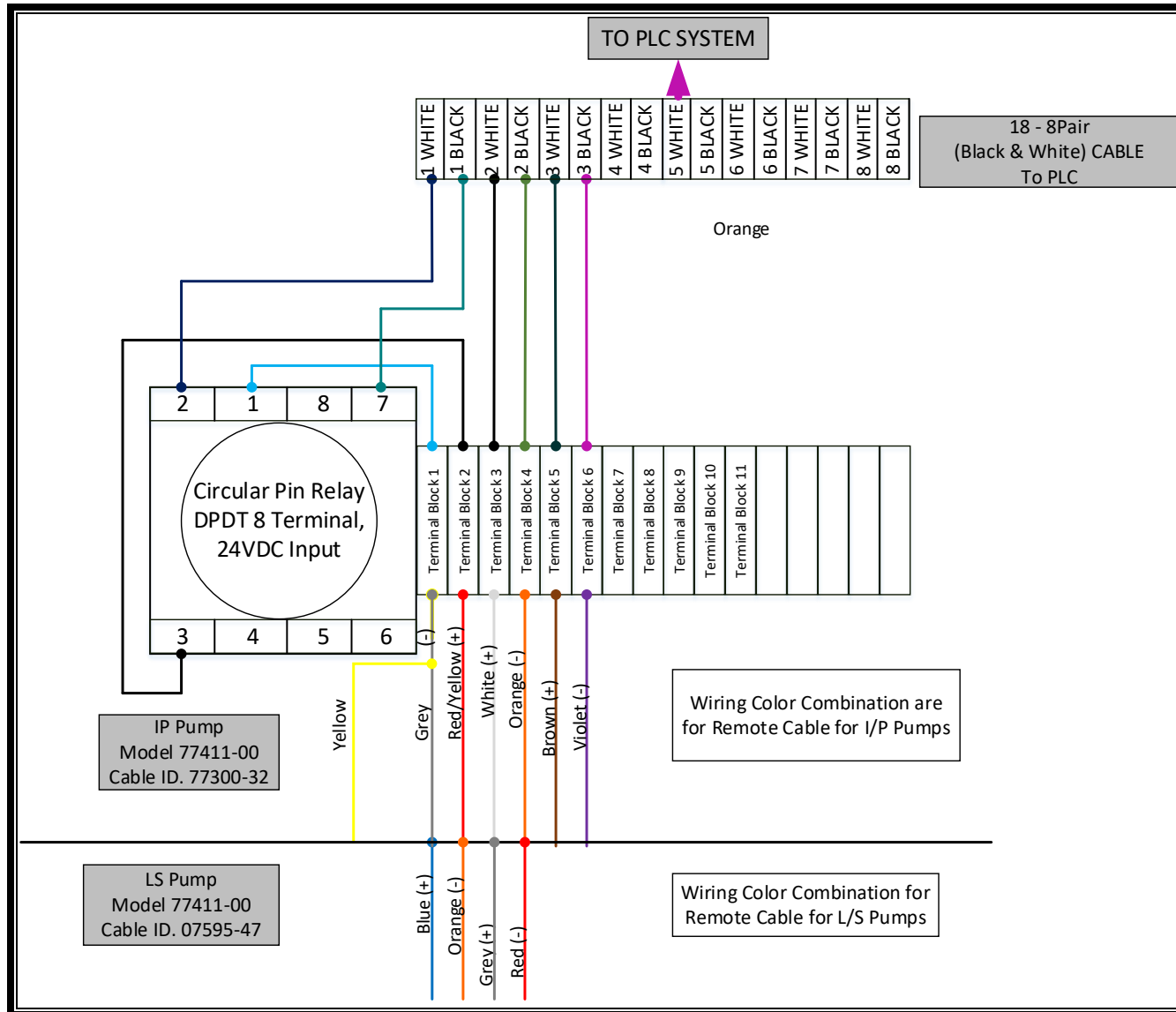
Figure 3.11. Junction box wiring diagram for sensor pump relay and instrumentation.
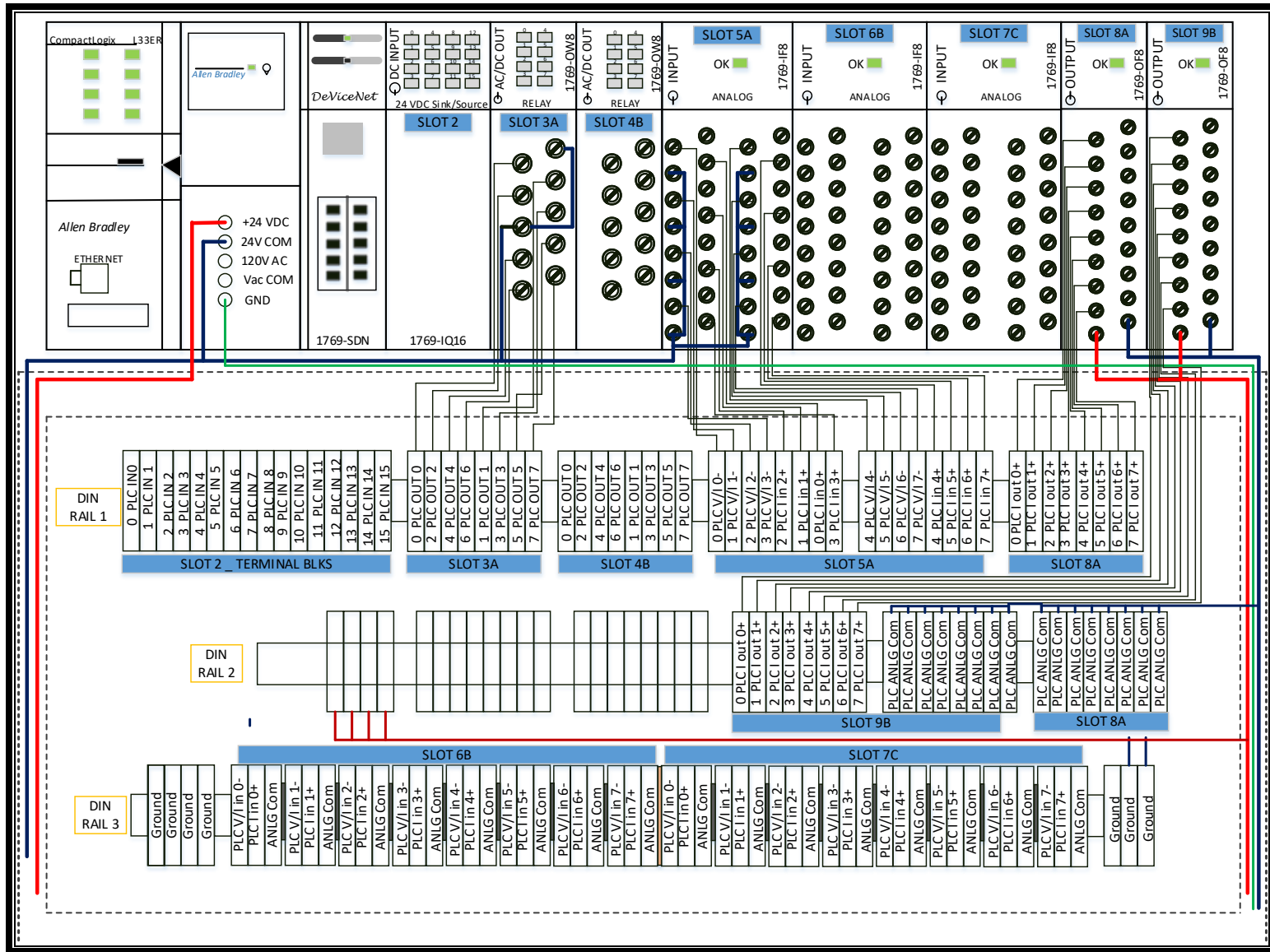
Figure 3.12. PLC wiring diagram in enclosure box (upper section).
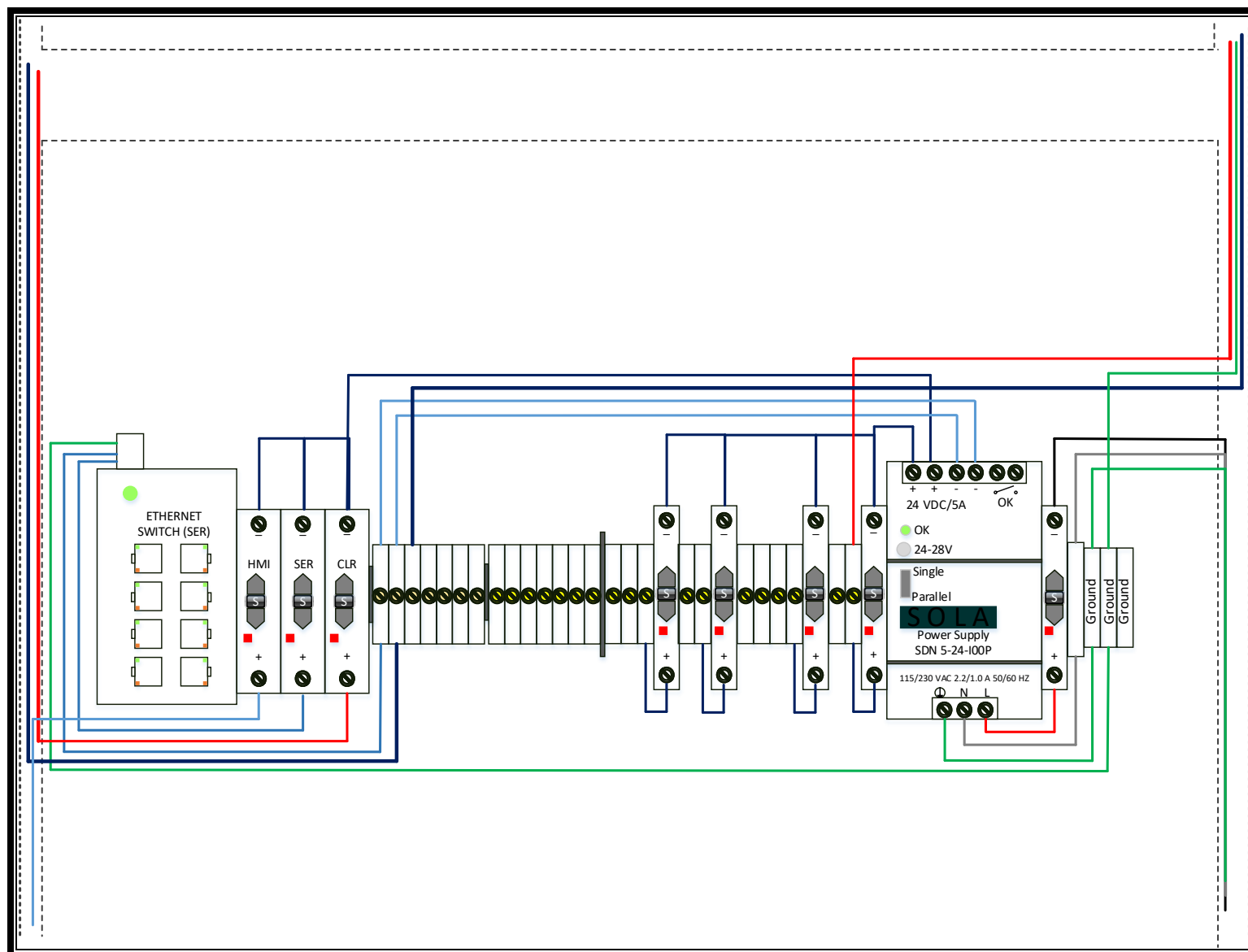
Figure 3.13. PLC wiring diagram for bottom section for control box.

Table 3.3. Sensors and Equipment mapping to I/O channels.

PART NO.	SENSOR/PUMP TYPE	INSTRUMENT DESCRIPTION	OUTPUT MODULE CHANNEL(START/STOP)	OUTPUT MODULE CHANNEL (SPEED CONTROL)	INPUT MODULE CHANNEL (RPM FEEDBACK)
EW-77411-00	I/P Pump - 1	Solvent Extraction Organic Feed Pump	Local 3:O.Data.0	-	Local 6:I.Ch0Data
EW-77411-00	I/P Pump - 2	Leach Tank 13 Outflow Pump into TK10/TK3	Local 3:O.Data.1	Local 8:O.Ch1Data	Local 6:I.Ch1Data
EW-77411-00	I/P Pump - 3	Thickener Tank 12 to Slurry Tank 14	Local 3:O.Data.2	Local 8:O.Ch2Data	Local 6:I.Ch2Data
EW-77411-00	I/P Pump - 4	Thickener Tank 2 to Leach Tank 6	Local 3:O.Data.3	Local 8:O.Ch3Data	Local 6:I.Ch3Data
EW-77411-00	I/P Pump - 5	Solvent Extraction Feed Pump	Local 3:O.Data.4	Local 8:O.Ch4Data	Local 6:I.Ch4Data
EW-77411-00	I/P Pump - 6	Raffinate Tank 15 to Waste Treatment Tank 19	Local 3:O.Data.5	Local 8:O.Ch5Data	Local 6:I.Ch5Data
EW-07528-10	L/S Pump - 1	Sulfuric Acid Controlled Pump into pH Neutralization Tank 4	Local 3:O.Data.6	Local 8:O.Ch0Data	-
EW-07528-10	L/S Pump - 2	Ascorbic Acid Pump from Tank 16 into Eh Control Tank 16	Local 3:O.Data.7	Local 8:O.Ch7Data	-
EW-	L/S Pump - 3	Sodium Hydroxide (NaOH)	Local 4:O.Data.0	Local	-

07528-10		Pump into pH Neutralization Tank 4		9:O.Ch0Data	
EW-07528-10	L/S Pump - 4	Solvent Extraction Scrubbing System Acid Feed Pump	Local 4:O.Data.1	Local 9:O.Ch1Data	-
EW-07528-10	L/S Pump - 5	Sodium Hydroxide (NaOH) Contolled Feed Pump into Waste Treatment Tank 19	Local 4:O.Data.2	Local 9:O.Ch2Data	-
EW-07528-10	L/S Pump - 6	Solvent Extraction (SX) Stripping System Acid Feed Pump	Local 4:O.Data.3	Local 9:O.Ch3Data	-
EW-07528-10	L/S Pump - 7	SX Circuit Saponification System Acid Feed Pump	Local 4:O.Data.4	Local 9:O.Ch4Data	-
EW-07528-10	L/S Pump - 8	Strip Solution Bleed Feed Pump from SX System to Cleaner Circuit	Local 4:O.Data.5	Local 9:O.Ch5Data	-
EW-07528-10	L/S Pump - 9	SX System Saponification Bleed into Waste Treatment Tank 19	Local 4:O.Data.6	Local 9:O.Ch6Data	-
EW-07528-10	L/S Pump - 10	Sulfuric Acid Controlled Feed Pump into Leaching Tank 6	Local 4:O.Data.7	Local 9:O.Ch7Data	-
S8000CD	pH Electrode	Sensorex pH Sensor for Control in Neutralization Tank 4	-	-	Local 5:I.Ch0Data

S8000CD	pH Electrode	pH Electrode for Control in Leaching Tank 6	-	-	Local 5:I.Ch1Data
S8000CD	pH Electrode	pH Control Sensor for Waste water Treatment in Tank 19	-	-	Local 5:I.Ch2Data
S8000CD-ORP	ORP Sensor	ORP Sensor probe for Eh control in Reduction Tank 3	-	-	Local 5:I.Ch3Data
AB 873P-D30AI-2500-D4	Ultrasonic Sensor	Ultrasonic sensor for Level Control in Raffinate Tank 15	-	-	Local 5:I.Ch4Data
AB 873P-D30AI-2500-D5	Ultrasonic Sensor	Ultrasonic sensor for Level Control in Leachate Tank 13	-	-	Local 5:I.Ch5Data

Table 3.4. Pumping system-wiring pairs to PLC.

		WIRING PAIRS	
FUNCTION	EQUIPMENT/SENSOR TYPE	(+) PIN COLOR	(-) PIN COLOR
Start/ Stop (4-20mA)	I/P Pumps	Red/Yellow	Grey+Yellow
Speed Control (4-20mA)		White	Orange
Output Speed Feedback (4-20mA)		Brown	Violet
Start/ Stop (4-20mA)	L/S Pumps	Blue	Orange
Input: Speed Control (4-20mA)		Grey	Red

3.2.3   Instrumentation

This section presents the different equipment and instrumentation sensors selected for use at the pilot plant. Selection is based on their low price, suitability and reliability for industrial chemical processes. The Allen Bradley based PLC system support both digital and analog inputs and outputs, with the controls engineer or operator able to configure the channels by physical wire mapping and through RSLogix 5000 programming platform. Four main process parameters were controlled by the PLC in the pilot plant: pH/ORP, temperature, flow control, and liquid level. A description of the instrumentation used to control each process parameter is provided below. Since budget is of concern in the operation, quality instrumentation at low cost remains a primary consideration.

Fluid Level control – Several methods are available to determine fluid levels of a holding tank. Level control was achieved with an ultrasonic single analog sensor (Model AB 873P-D30AI-2500-D4, Rockwell Automation) Figure 3.14. The sensor was secured onto the middle of Tank 13 and 15 and used to send a sound wave which bounces back on the material surface and calculates the distance based on the time the wave returns. This sensor is suitable for the pilot plant holding tank level control based on the operational design and method (more information in Appendix).

Figure 3.14. Ultrasonic sensors for level control.

Heating control – Heating and temperature measurement was done using a 4000-watt single phase only heater (Model HX 4229-P2, Process Technology, USA) with a thermocouple (Type K) located away from the tank bottom and sides. The fluoropolymer heater has built in thermal protector and grounded element. It has corrosion and chemical resistant properties. The heater was secured into the tanks at approximately 7 inches from the center through a duct made from the tank lid to heat the leachate as shown in Figure 3.15. The heater is used in combination with a conductive liquid level probe (LC Series Model LC2H12, Process Technology) pre-wired into an automated controller. The heater is controlled using thermocouple readings feed to a controller (Model DE502-P2-LC, Process Technology). Figure 3.16 shows the heat control components used in the leaching circuit. The conductive level control adds protection against damage and tank fires associated with low-level conditions

Figure 3.15. Heating control unit mounted on a leaching tank.
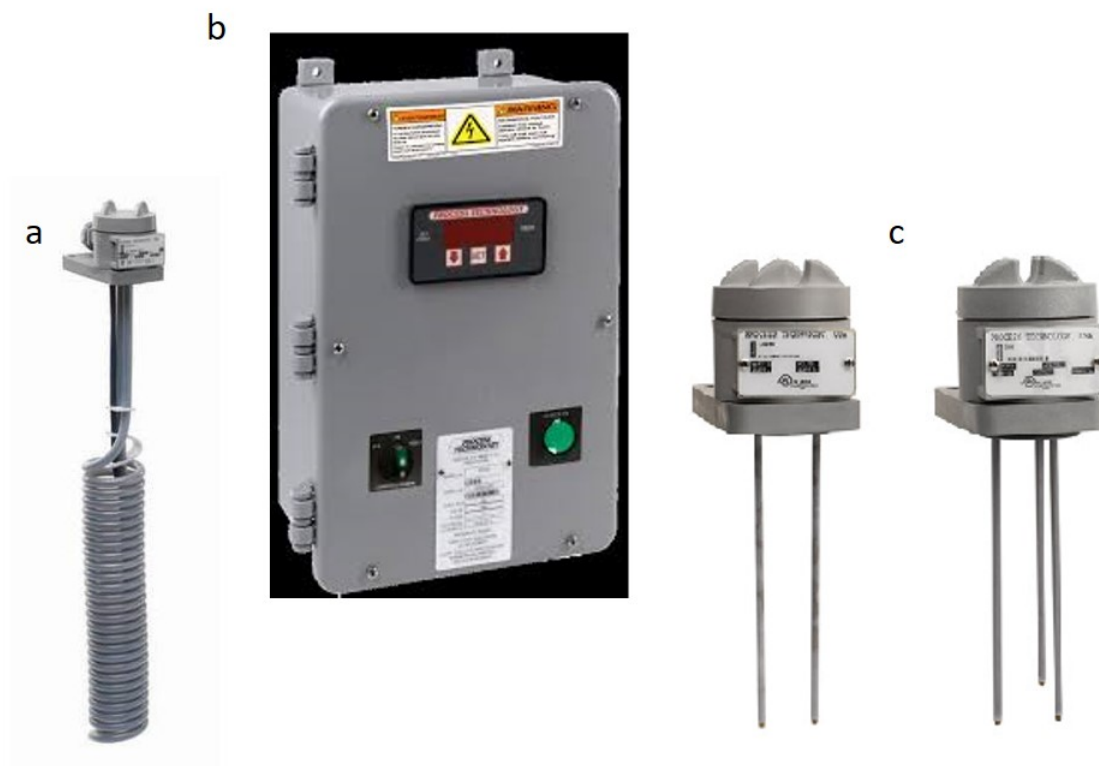
Figure 3.16. Heating control components (a) HX4429-P2 (b) DE502-P2-LC (c) LC2H12

pH/ORP Control – An industrial quick-change replacement pH and ORP cartridge (Model S8000CD and S8000CD-ORP, Sensorex, Garden Grove, CA) was used to measure and control the pH in Tanks 4, 6, 19 and the reduction potential in Tank 3 respectively. Whilst the pH/ORP electrode is configured for submersion use, it is also assembled with a 4-20mA blind electronic module transmitter (Model: EM802-PH, and EM802-ORP Sensorex), an electrode adapter with built in ATC (automatic temperature control) with solution ground pin (Model: EA899TC, and EA899 Sensorex, USA) and coaxial cable (Model S853/20/TL, Sensorex). As shown in Figure 3.17, the electrode is loop powered by the PLC and used for tank solution neutralization using PID control method. A pump is used to add acid or base to the controlled system.

Figure 3.17. Sensorex S8000CD pH/ORP electrode.

Pump Control – A large portion of the pilot-plant control system involves the handling of pumps. Most of these pumps such as the Tank 5 to SX mag drive centrifugal pump (Model WMD-30RLZT-115, Iwaki), are easily controlled by a remote relay level controller (Model LC41-1001, Cole Parmer) activated by two capacitance level switch sensors (Model LP15-1405, Cole Parmer). Other pumps such as the recirculation pumps and inlet pumps (Masterflex I/P 77411-00 or L/S 07528-10, Cole Parmer) in Figure 3.18 need to have variable flowrate to maintain system level and controls at steady state.

Constant relay ON/OFF switching would be inefficient hence, require analog 4-20mA signal for variable speed control. Analog signaling to the pump drive from the PLC would require a DB-9 external control connector (Cole Parmer EW-07595-45). PID controller adjusts the wired pumps (see Figure 4.8 or Table 4.4) output speed (RPM) during operation.
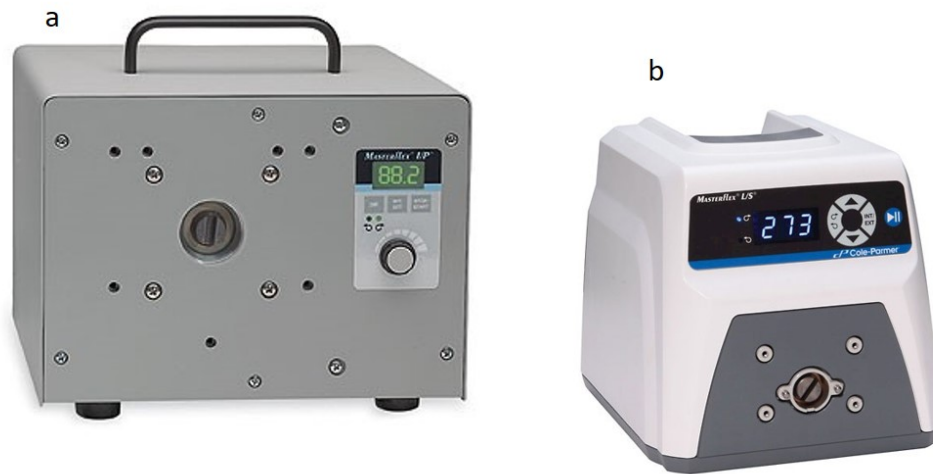
Figure 3.18. Masterflex pumps (a) I/P pump (b) L/S variable speed pump.

3.3   Preliminary Process Control Development

This section involves the investigation and implementation of six-sigma methods to develop a PLC based automated control system. Benchmarking activities and discussions was conducted to gather significant information on critical processes in the pilot plant and variables critical to operation while exploring control theories.

An important phase of the control system development for safety and quality planning is the development of a control plan (see Appendix 1). The Control Plan is a written description of the systems used in controlling leaching process parts. The control plan is intended to document and communicate the initial plan for leaching process control. Subsequently, it guides the operator or any technical staff in how to control the process to ensure the achievement of control objective. It undergoes constant update as control methods and measurements tools are evaluated and improved. As part of a general rule to establishing an effective control plan for the leaching control, the team utilized the basic understanding of the process and available information to develop and initial control plan such as:

- Process flowsheet
- Design/process failure mode and effects analysis (see Appendix 2)

- Previous learnings
- Team expertize or experience with process

Benefits of a control plan includes reducing waste and ensuring improvement in product quality, communicating changes and process improvement.

3.3.1 Leaching and Wastewater Circuit control and loop tuning

In order to recover REEs from coal based sources in an effective and efficient approach, a pilot scale operation was performed to examine and change the circuit operation for leaching, wastewater treatment, and solvent extraction rougher processes. As in Figure 3.19, a leaching test was operated continuously for 10 hours. Tanks 6 and Tank 7 was used to perform a two stage solid acid leaching process to achieve the maximum leaching performance at a retention time of 4 hours.
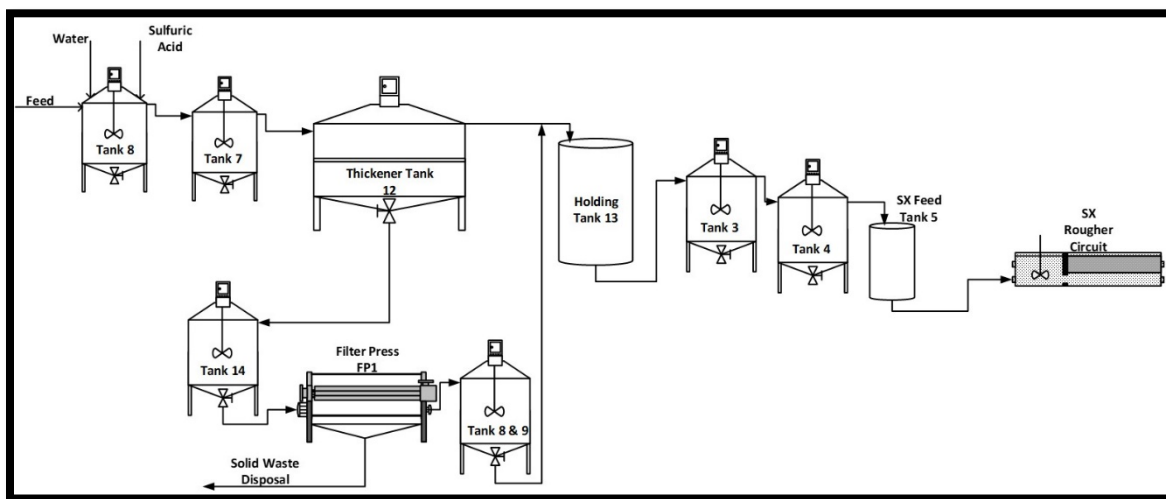
Figure 3.19 Leach circuit operation diagram

Through automation, the pH value of the acid leaching solution in Tank 6 was controlled with the industrial pH electrode (Model S8000CD, Sensorex) and peristaltic variable speed pump (Masterflex L/S 07528-10, Cole Parmer) that feeds in sulfuric acid (H_2SO_4) by the PLC via PID methodology. Similarly, Figure 3.20 shows the setup of PLC-based pH control of the wastewater in Tank 19 with a pH sensor and peristaltic pump that feeds sodium hydroxide. The control applies trial and error loop tuning method for the PID

algorithm through the RSLogix 5000 ladder logic programming. As a safety measure, all operators are required to take note of all trainings prior to operation.
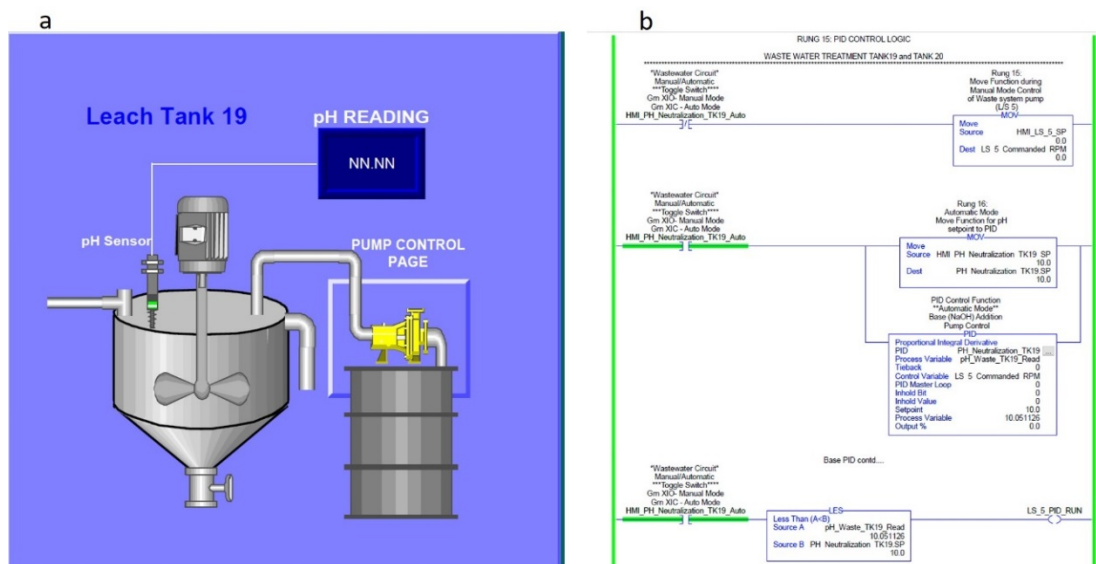
Figure 3.20 Wastewater system operation diagram (a) pH control (b) PID logic program.

To operate the leaching process, both 100 gallon by volume Tank 6 and Tank 7 were filled with 350 liters of 1.2M sulfuric acid and heated (HX 4229-P2, Process Technology) to 75°C. Through a screw feeder system, coal material conveyed at a rate of 50 lbs. per hour was fed into Tank 6 alongside water at a controlled flowrate of 1 gallon per minute, achieving 20% concentration. The automated pH control system was started and the operator recorded slurry weight and pH value from collected slurry samples every 2 hours. From the RSLogix 5000 ladder programming, automatic PID control was implemented with trial and error loop tuning methodology to achieve best control. To reduce electrical disturbances to the readings by the pH sensor, an array-based moving average function block over 12 seconds was used to calculate the final process variable (see Appendix 3).

3.3.2 Solvent Extraction operation

The solvent extraction circuit unit was originally designed and built by SX Kinetic, Canada. Before operating SX units, some equipment parts were replaced and some minor

modifications were implemented.A simplified operational flow diagram of the SX rougher unit can be seen in Figure 3.21 below.
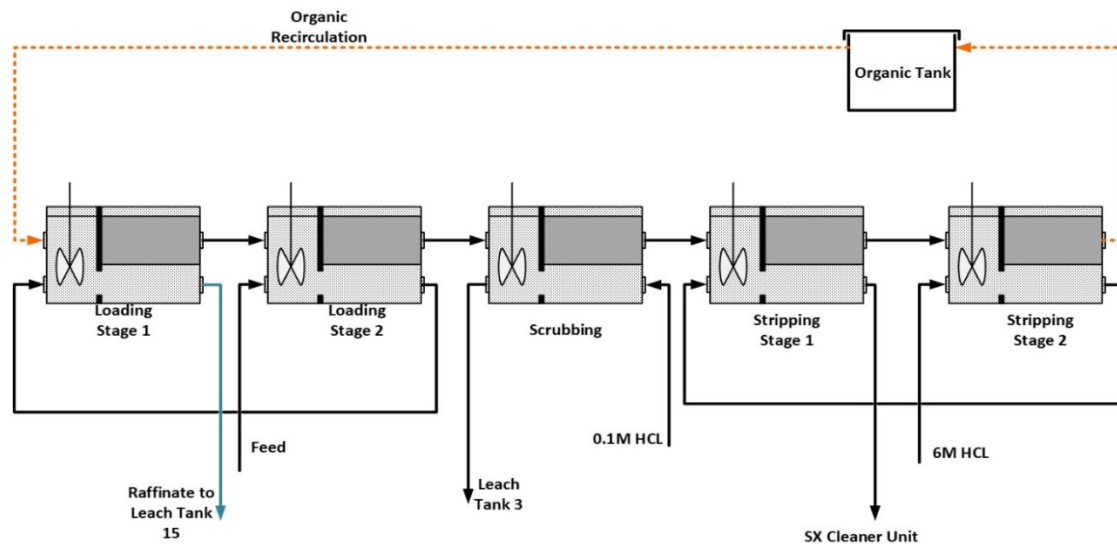
Figure 3.21 SX Rougher circuit operation diagram.

Pretreatment of SX feed starts in Tank 3 and Tank 4 (see Figure 3.1). Using the same automatic PLC-based PID control appraoch, iron contaminant in the leachate generated from acid leaching is controlled using an industrial ORP sensor (S8000CD-ORP) and peristaltic pump that continuously doses ascorbic acid solution into Tank 3 to reduce ferric iron into ferrous (Figure 3.19). In other words, leach solution entering Tank 3 has a higher ORP value (more ferric), hence ascorbic acid lowers the ORP value of solution (indicative of near complete reduction of iron). The ORP value of the solution was reduced and maintained at 300 mV. Likewise the pH value of the reducing stage solution in Tank 3 decreased.

The solution overflows into neutralization Tank 4 (Figure 3.1) were the pH value of the solution was controlled with sodium hydroxide by applying autotuned PID control (see Appendix 4). The solvent extraction rougher operational setup shown in Figure 3.21 consist of two loading stages, a scrubbing stage, and two stripping stages. The two latter stages were built to internally recirculate the acqueous stream. Startup operation includes

- Start organic recirculation pump at fixed speed (22 l/min).

- Allow system to run for approximately 30 minute before turning off organic recirculation pump.

- Start loading stage feed at fixed pump flow rate (2 liters per minute)

- Start scrubbing pump to fill mixer-settler tank with 0.1 molar hydrocloric acid at fixed pump speed of 20rpm.

- Fill stripping mixer-settler tank with 6 molar hydrocloric acid and fixed pump speed of 18 rpm.

Samples taken from stripping and scrubbing stages are analysed. Further treatment of the strip solution in rougher stage is achieved at the cleaner solvent extraction circuit.

3.3.3 Cleaner Unit operation

The continuous SX cleaner circuit consist of 6 mixer (270ml)–seetler (1050ml) glass unit, as shown in Figure 3.22. The cleaner SX unit setup consist of 3 stages of loading and 3stages of stripping. Operational startup is similar to that of the SX rougher circuit. The feed to the cleaner unit is a bleed from the SX rougher stripping stage. Using PID scheme, the feed pH value is maintained at 0.9 whiles operating the organic recirculation pump at a predetermined speed.

Figure 3.22 Solvent Extraction cleaner unit.

The stripping stage mixer-settler was filled with 6 molar hydrocloric acid while running the agitators in each unit.The raffinate from the loading step is recirculated to the SX rougher circuit and bleed solution from the cleaner stripping stage is transferrd for precipitation

3.3.4   GUI-HMI Design

The graphics user interface (GUI) of the pilot plant is displayed on the PLC's HMI. Design of HMI was achieved with a computer-based FactoryTalk View software by AB Rockwell Automation (Site Edition –SE) For the operator on the plant floor, the HMI is made available on a 15 inches color touch screen (AB 2711P-T15C21D8S) mounted on the PLC enclosure box as shown in Figure 3.23. Different display screens have been created along with the ladder logic programming of the various parts of the plant system.

Figure 3.23 Touch screen for HMI display.

The HMI main screen presents the operator (user) with an overview of the pilot plant leaching process diagram and displays three specific push buttons linking to different screens (Figure 3.24). Similarly, the second overview screen displays the status operation

of the wastewaster circuit (Figure 3.25). The button tabs allow the user to navigate through screens.
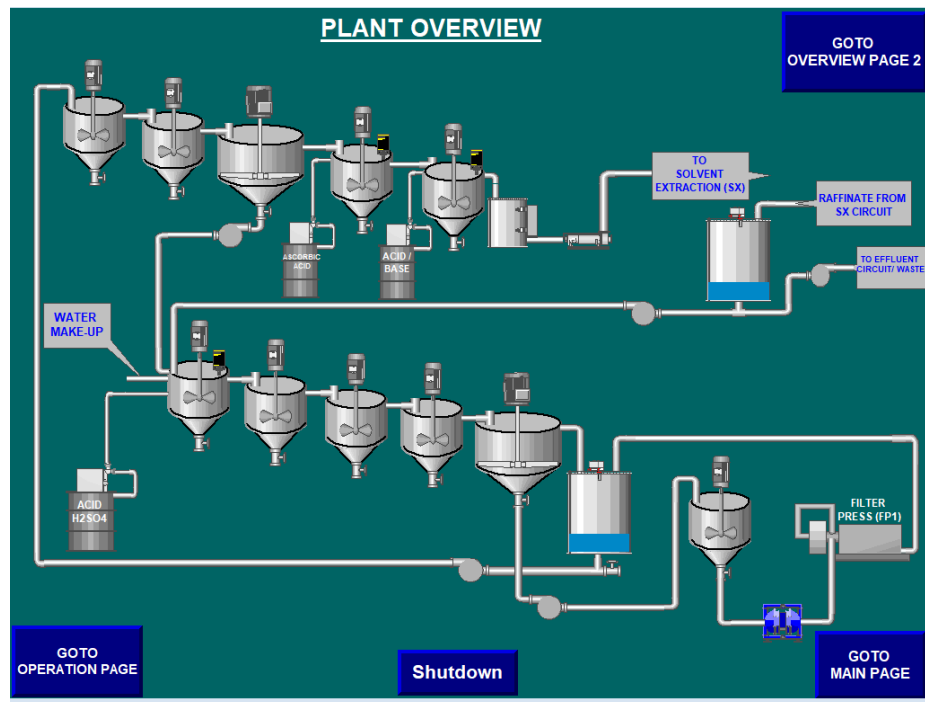
Figure 3.24. HMI main screen for leaching process.
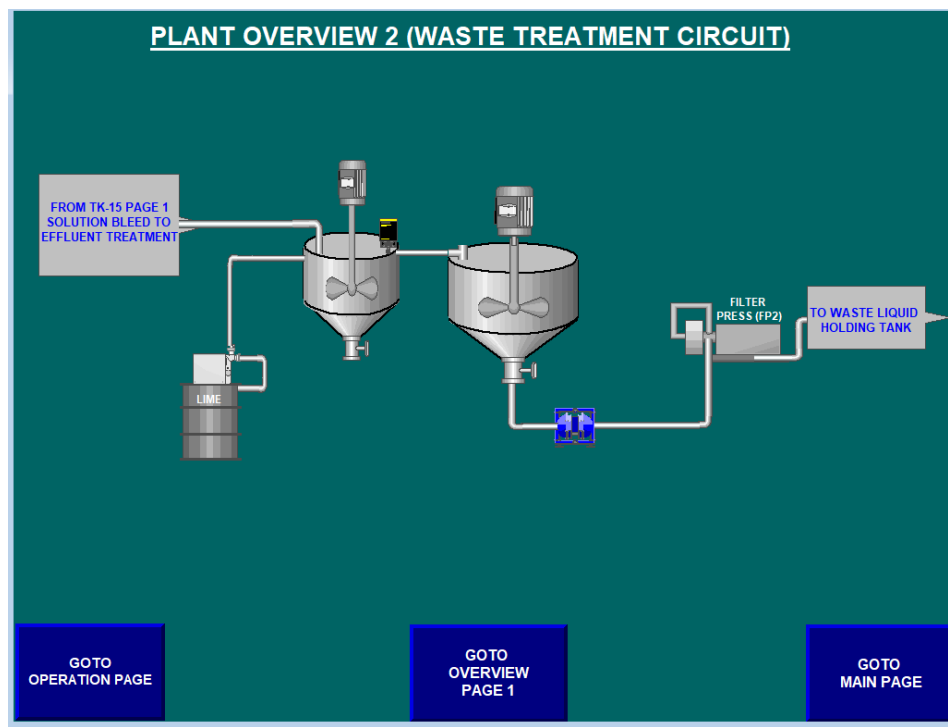
Figure 3.25. HMI wastewater circuit overview.

The next panel display present the user with the operational modes for the critical variables and displays the current date and time. There, the current status of each control operation is shown and presents monitoring possibilities (Figure 3.26) and gives a brief status description.
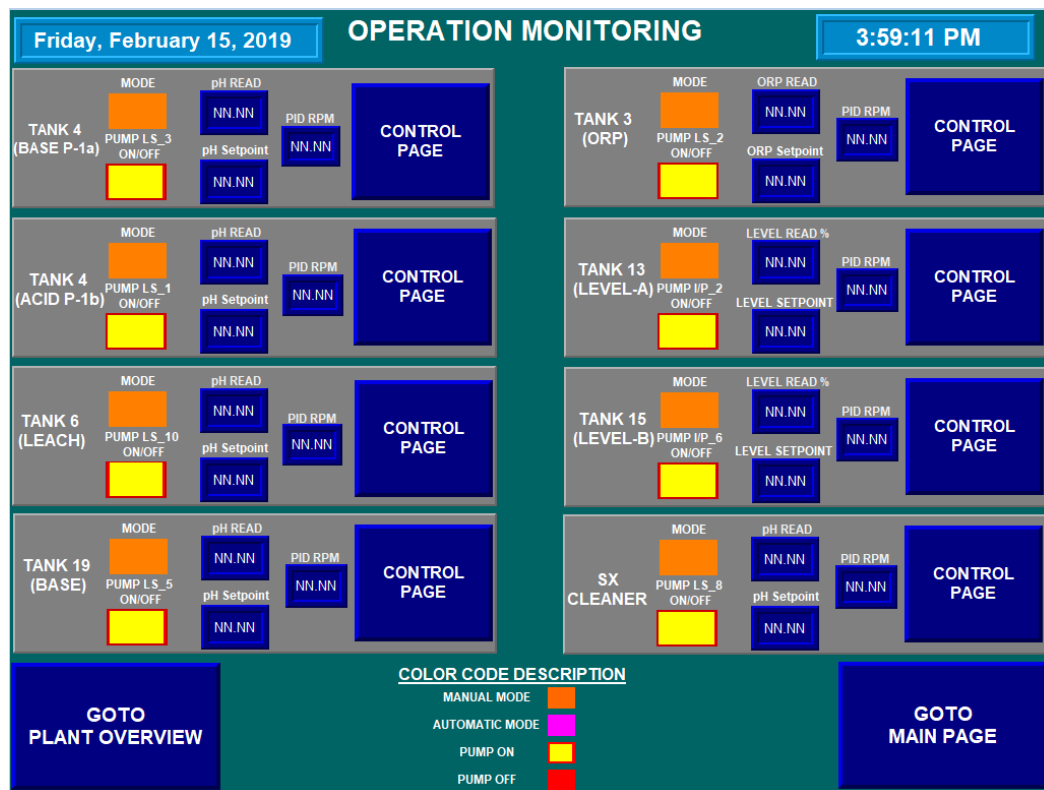
Figure 3.26 HMI operation monitoring display.

A push button (control page) can be seen for each tank, which presents a new screen for controls. Control page panel for Tank 6 and Tank 3 for example shows the graphical control for the pH and ORP along with their respective pump system (Figure 3.27). When pump images are pushed, it is possible to access the relevant trends associated with the control.
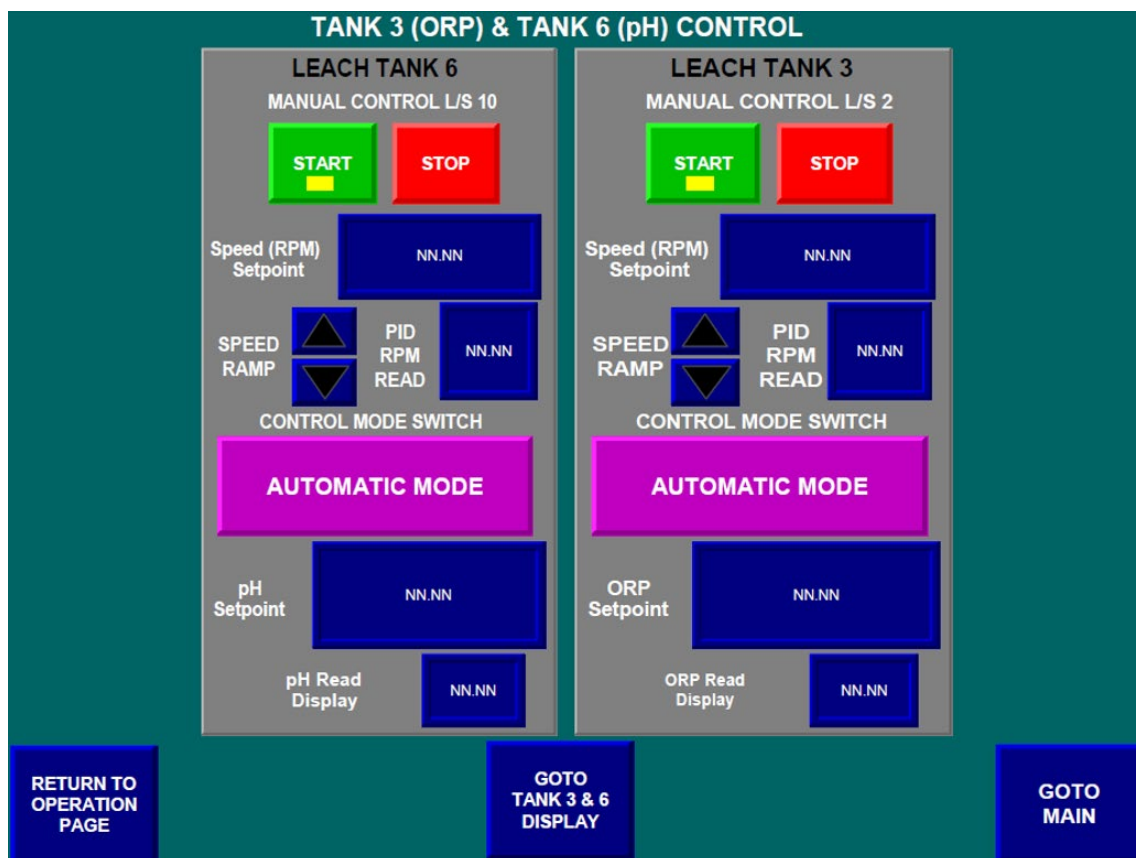
Figure 3.27 HMI pH and ORP control screen.

CHAPTER 4. PERFORMANCE MONITORING TRENDS

Chapter 4 presents the initial performance results specific to leaching pH control process for Tank 6 as described in chapter three. Discussion and basic conclusions in the results is presented. Trends shown here have been created to best describe the current PID control performance with supporting data included.

4.1 Trends

The HMI is also used as a monitoring tool for present and past data of analog and digital types. These trend graphs are designed and programmed using the Factory Talk View SE software. Based on operators need; the HMI trend displays control performance at different times. With different number of process variables being maintained within specified limits in order for the plant to operate smoothly, routine process monitoring will ensure that system performance satisfies the operating objectives of the plant.

Statistical process control was evaluated to ensure that the process operates efficiently. Figure 4.1, and Figure 4.2 show control charts demonstrating the performance of leaching experimentation pH for different case scenarios at specific set points. In each experimental case, the leaching temperature condition was maintained at 75℃ and constant feed rate. Table 4.1, and Table 4.2 show the test process data for Case 1, and 2 at pH set points 2.0, and 2.3 respectively.

Tank 6 leaching pH monitoring test.

The representative trend in Figure 4.1 is the performance of the leaching pH experiment at setpoint 2.0 over a 1-second sample rate. For Case 1 test, the average pH recorded was 1.96 with an estimated error value of 0.042 based on trial and error tuning setup. By estimating the standard deviation (σ) of the sample data, the control limits were computed to determine the $\pm 3\sigma$ control limits (UCL - upper control limits and LCL - lower control limit) to characterize the system.
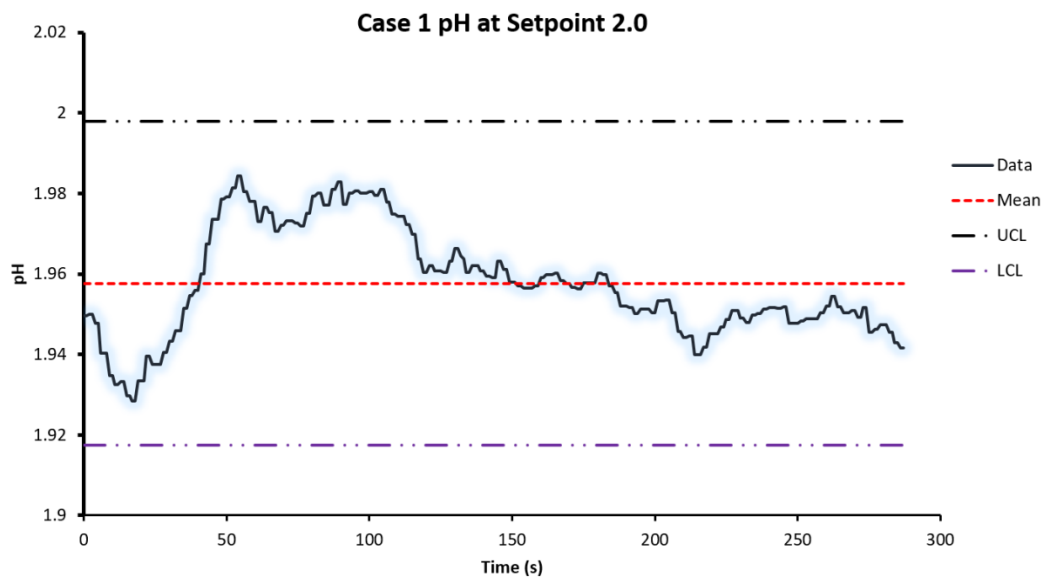
Figure 4.1. Trend of leaching test pH control.

Table 4.1. Average pH data recorded.

Initial pH Setpoint	Mean pH value	Standard Deviation (σ)	Upper Control Limit (UCL)	Lower Control Limit (LCL)	Error	Proposed pH setpoint
2.00	**1.96**	**0.0134**	**1.99**	**1.92**	**0.042**	**1.96**
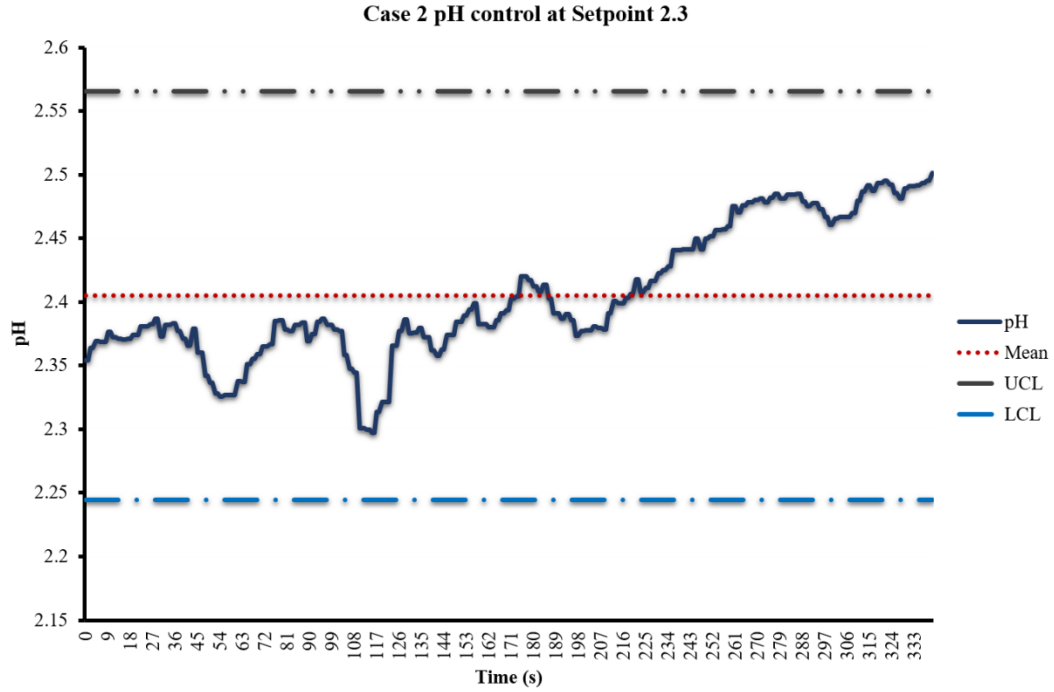
Figure 4.2. Leaching pH control performance.

Table 4.2. Mean pH value recorded for Case 2.

Initial pH Setpoint	Mean pH value	Standard Deviation (σ)	Upper Control Limit (UCL)	Lower Control Limit (LCL)	Error	Proposed pH setpoint
2.30	2.40	0.054	2.57	2.24	0.10	2.20

The representative performance trend for Case 2 pH experimentation in Figure 4.2 at pH set point 2.30 recorded a mean pH value of 2.40. Similar to Case 1, the control limits ± 3σ were computed from the estimated sample data standard deviation. Based on this result, the typical process variation can be inferred to detect any anomalous or out of control conditions. The small variability of data about the mean value for both cases is attributed to common electric noise associated with adjacent motor drives. Tuning of the PID will consequently help improve small deviations about the mean.

CHAPTER 5. CONCLUSION AND FUTURE WORK RECOMMENDATION

5.1   General Conclusion

The main goal for the development of a PLC-based process control system is to operate the pilot plant leaching and solvent extraction circuit safely, accurately, and at the lowest cost for the recovery of coal-based rare earth elements (REEs).

Literature review on leaching, solvent extraction, and process control assisted in a more complete understanding of mineral recovery processes and the different implementation methods of process control. The implemented Six Sigma techniques (control plan, FMEA, etc.) were used successfully to scope the process and provided valuable knowledge and understanding towards safe design and development of process controls.

Based on the experimentation, logic programming testing, system tuning (Trial and error method) and debugging the pilot-scale process control PLC system and logic described in this research work was able to operate and successfully control critical process variables (pH, ORP, level, and flow) in the recovery of REEs.

5.2   Recommendations

This project hopes to provide useful and effective method to the design and build of industrial process control for the operation and mineral recovery leaching and solvent extraction processes. As part of ongoing research, the following recommendations are suggested for future work.

1. Further system tuning to fully regulate process variables and fulfill the process optimization objective.

2. Frequent calibration of field sensors and instruments will be performed to improve equipment life and performance.

3. SCADA and supervisory level data will be used to perform advanced process control and statistical analysis.

4. Further sensor signal testing with analog filters or signal conditioners will help reduce variability of sensor data attributed to common electric noise.

APPENDIX 1 LEACHING PROCESS CONTROL PLAN

<td colspan="10" align="center">**Control Plan**</td>
Control Plan Number: 001-Leach Process Control Design		Control Plan Owner / Phone: Douglas (859)684-1690				Date Original: 05/10/2017		Date Revised: NA	
Process: REE 1/4 hr Pilot Plant		Team: Douglas Ado, Josh Werner, Bob Braton, Jacob Gill				Revision Note			
Description: Operation of Leach Circuit		Approval Date:							

Process Number	Process Name/ Description	Tank, Device, Equipment	Characteristics			CTQ	Methods					Reaction Plan
			No.	Inputs	Output		Product/Process Specification/Tolerance	Evaluation / Measurement Technique	Sample		Control Method	
									Size	Freq.		
1	Leaching	TK-10	10a	Filter Cake Solid Mass		N	70-75 %	VT?	?	?	?	?
			10b	Filter Cake Liquid Mass		N	25-30 %	VT?	?	?	?	?
			10c	Filter Cake Liquid pH		Y	?	VT?	?	?	?	?
			10d	Filter Cake Feed Rate		Y*	~100lb hr	VT?	?	?	?	?
			10e	Inlet Flow Rate (P-13)		Y	4 - 8 (Target 6 LPM)	RPM	N/A	Continuous	Set Point / PLC	Operator Adjust
			10f	Inlet Flow Temperature		Y	None	N/A	N/A	N/A	N/A	N/A

			10g		Leaching Temperature	Y	75 ℃	Thermocouple	N/A	Continuous	Set Point / PLC	Operator Adjust
			10i		Residence Time	Y	See Sys-a	See Sys-a	See Sys-a	See Sys-a	See Sys-a	See Sys-a
			10j		pH	Y	TBD	pH Probe	N/A	N/A	See Set Point in TK-6	SOP
			10k		% Solids	Y*	1-20%	Weight of Known Volume	N/A	Hourly	P-13 Flow Rate	SOP
	Leaching	TK-1	1a	Inlet Flow Temperature		N	None	N/A	N/A	N/A	N/A	N/A
			1b	Inlet Flow pH		N	None	N/A	N/A	N/A	N/A	N/A
			1c		Tank pH	Y	None	pH Probe	N/A	Continuous	N/A	N/A
			1d		Tank Temperature	Y	None	Thermocouple	N/A	Continuous	Display	N/A
	Thickener	TK-2	2a	Inlet Flowrate (TK-1)		N	None	N/A	N/A	N/A	N/A	Adjust
			2b	Inlet Slurry Temperature		N	None	N/A	N/A	N/A	N/A	N/A
			2c	Inlet Slurry pH		Y	None	N/A	N/A	N/A	N/A	N/A
			2d	Flocculent ADD Rate		Y	Rate of Flocculent Addition	RPM of Flocculent Motor	N/A	Continuous	Set Point/ PLC	Operator Adjust
			2e	Rake Speed		N	TBD	VFD	N/A	Continuous	Set Point/ PLC	Operator Adjust
			2f		Settling	Y	None	See 2k	N/A	N/A	N/A	N/A

Tank	Step	Parameter	Y/N	Spec	Measurement		Frequency	Control	Action
		Rate							
	2g	Depth of Solids/ Slurry Underflow Rate	Y*	At Minimum Rake Height	Amps on TK-2 Motor	N/A	Continuous	RPM P-2 Slaved to Amperage on TK-2	Alarm on Over Amperage (Amps)
	2h	pH	Y	TBD	Digital Readout		Continuous	See Set Point on TK-6	SOP
	2i	Temperature	N	Thermocouple	N/A	Continuous	N/A	See Set Point on TK-10	SOP
	2j	Underflow Slurry Flowrate (P-2)		2 - 4 (Target 2.2 LPM)	RPM	N/A	Continuous	RPM P-2 Slaved to Amperage on TK-2	PLC Program Adjustment
	2k	Clarity of Gravity Overflow	Y*	TBD	?	?	?	See 2d	SOP
TK-3	3a	Ascorbic Acid Solution pH	N	None	N/A	N/A	N/A	N/A	N/A
	3b	Ascorbic Addition rate (P-16)	Y*	1 - 2 (Target 0.2 LPM)	RPM	N/A	Continuous	Set Point / PLC	See 3e
	3c	Flowrate of liquid feed (TK-2)	N	Gravity	N/A	N/A	N/A	N/A	N/A
	3d	Inlet Flow Temperature	Y	None	N/A	N/A	N/A	N/A	N/A
	3e	ORP	N	TBD	ORP Probe	N/A	Continuous	Set Point / PLC	Operator Adjust

										adjustment of 3b	
			3f	Tank Temperature	Y	None	Thermocouple	N/A	Continuous	Display	N/A
			3g	Residence Time	Y	None	N/A	N/A	N/A	N/A	Combination of P-13 and P-2 flow Rates
		TK-4	4a	Inlet Flow Rate TK-3	N	Gravity Overflow	N/A	N/A	N/A	N/A	N/A
			4c	Inlet Flow Temperature	N	None	N/A	N/A	N/A	N/A	N/A
			4d	Inlet Flow Rate NaOH (P-17)	Y	1-2 (Target 0.74 LPM)	RPM	N/A	Continuous	Set Point/ PLC	See 4e
			4e	pH	Y*	TBD	Digital Readout	N/A	Continuous	Set Point / PLC adjustment of 4d	Operator Adjust
			4f	Tank Temperature	Y	N/A	Thermocouple	N/A	Continuous	Display	N/A
		TK-5	5a	pH	N	N/A	N/A	N/A	N/A	4e	N/A
			5b	Inlet Temperature	Y	N/A	N/A	N/A	N/A	N/A	N/A
			5c	Tank Level	Y	Between High and Low Level	High / Low Sensor	N/A	Continuous	Controller to P-5 / Tank	Alarm if over High Level for

				Parameter		Y/N	Description	Sensor			Level Display	more than X seconds
			5d	Fe2+ to FE3+		Y*	No FE3+	ORP	N/A	Continuous	Air Tight	Recirculate to TK-3
			5e	Residence Time		Y	None	N/A	N/A	N/A	N/A	Recirculate to TK-3 on start up
			5f	Tank Temperature		N	N/A	N/A	N/A	N/A	N/A	N/A
			5h	Outlet Flowrate (P-5)		Y*	On/Off	High / Low Sensor	N/A	Continuous	Level Control in TK-5 / PLC	Alarm if over High Level for more than X seconds
			5i	Outlet Flow pH		N	None	N/A	N/A	N/A	4e	N/A
		TK-6	6a	Inlet Leachate Flow (P-2)		Y	See 2j	See 2j	See 2j	See 2j	See 2j	See 2j
			6b	Inlet Solution Flow (P-15a)		Y	See 15c	See 15c	See 15c	See 15c	See 15c	See 15c
			6c	Inlet Flow rate Water Make-Up		Y*	Near Constant Water Addition	RPM	N/A	Continuous	Slaved rate of P-15b with Time average override of summation TK-13 and TK-	Plc programming

											14 via PLC	
			6d	Inlet Flow Temperature		N	None	N/A	N/A	N/A	N/A	N/A
			6e	Inlet Flow pH		N	None	N/A	N/A	N/A	N/A	N/A
			6f	Inlet Flowrate Acid (P-1)		Y	0 - 2 (Target 0.18 LPM)	RPM	N/A	Continuous	Set Point/ PLC	Operator Adjust
			6g		Tank pH	Y*	0 - 1	pH Probe	N/A	Continuous	Set Point/ PLC	Operator Adjust
			6h		Tank Temperature	Y*	60 - 75 ℃	Thermocouple	N/A	Continuous	Set Point/ PLC	Operator Adjust
			6i		Residence Time	Y	See Sys-a	See Sys-a	See Sys-a	See Sys-a	See Sys-a	See Sys-a
		TK-7 to TK-10	7a	Inlet Flow Temperature		N	None	N/A	N/A	N/A	N/A	N/A
			7b	Inlet Flow pH		N	None	N/A	N/A	N/A	N/A	N/A
			7c		Tank pH	Y	None	pH Probe	N/A	Continuous	N/A	N/A
			7d		Tank Temperature	Y	60 - 75 ℃	Digital Readout	N/A	Continuous	Set Point/ PLC	Operator Adjust
			7e		Residence Time	Y	See Sys-a	See Sys-a	See Sys-a	See Sys-a	See Sys-a	See Sys-a

	Thickener	TK-12	12a	Inlet Flowrate (TK-9)		N	None	N/A	N/A	N/A	N/A	Adjust
			12b	Inlet Slurry Temperature		N	None	N/A	N/A	N/A	N/A	N/A
			12c	Inlet Slurry pH		Y	None	N/A	N/A	N/A	N/A	N/A
			12d	Flocculent ADD Rate		Y	Rate of Flocculent Addition	RPM of Flocculent Motor	N/A	Continuous	Set Point/PLC	Operator Adjust
			12e	Rake Speed		N	TBD	VFD	N/A	Continuous	Set Point/PLC	Operator Adjust
			12f		Settling Rate	Y	None	See 12k	N/A	N/A	N/A	N/A
			12g		Depth of Solids/Slurry Underflow Rate	Y*	At Minimum Rake Height	Amps on TK-12 Motor	N/A	Continuous	RPM P-10 Slaved to Amperage on TK-12	Alarm on Over Amperage (Amps)
			12h		pH	Y	TBD	Digital Readout	N/A	Continuous	See Set Point on TK-6	SOP
			12i		Temperature	N	Thermocouple	N/A	Continuous	N/A	See Set Point on TK-9	SOP
			12j		Underflow Slurry Flowrate (P-10)		2 - 4 (Target 2.2 LPM)	RPM	N/A	Continuous	RPM P-10 Slaved to Amperage on TK-12	PLC Program Adjustment
			12k		Clarity of Gravity Overflow	Y*	TBD	?	?	?	See 12d	SOP

Tank	ID	Parameter	Parameter	Y/N						
TK-13	13a	TK-12 Overflow		N	Gravity Overflow	N/A	N/A	N/A	N/A	N/A
	13b	Flow from FP-1		Y	Variable From Filter press	N/A	N/A	N/A	N/A	N/A
	13c	pH		N	None	N/A	N/A	N/A	See Set Point on TK-6	N/A
	13d	Inlet Temperature		N	None	N/A	N/A	N/A	N/A	N/A
	13e		Tank Temperature	Y	None	N/A	N/A	N/A	N/A	N/A
	13f	Outlet Flowrate (P-13)		Y	4-8 (Target 6 LPM)	RPM	N/A	Continuous	Set by operator / PLC	SOP
	13g		Outlet pH	Y	None	N/A	N/A	N/A	N/A	N/A
TK-14	14a	Mass of Solids		N	N/A	N/A	N/A	N/A	N/A	N/A
	14b	Inlet Temperature		N	None	N/A	N/A	N/A	N/A	N/A
	14c	Slurry Flowrate (P-10)		Y	See 12j	See 12j	See 12j	See 12j	See 12j	See 12j
	14d		pH	N	None	N/A	N/A	N/A	N/A	N/A
	14e		Tank Temperature	N	None	N/A	N/A	N/A	N/A	N/A
	14f		Slurry Flowrate (P-14)	Y*	See FP1c	See FP1c	See FP1c	See FP1c	See FP1c	See FP1c

						Y/N						
		FP - 1	FP1a	Inlet SX Raffinate (P-14)		Y*	TBD	?	N/A	?	Auto ramping pressure by filter filling followed by need to cycle	Operator cycle
			FP1b	Inlet Temperature		N	None	N/A	N/A	N/A	N/A	
			FP1c		Filter Cake Solid Mass	Y	70-75%	TBD	TBD	TBD	TBD	TBD
			FP1d		Filter Cake Liquid Mass	Y	25-30 %	TBD	TBD	TBD	TBD	TBD
			FP1e		pH	N	None	N/A	N/A	N/A	N/A	N/A
		TK-15	15a	Spent Scrub/ Raffinate Flow Rate (SX_P-6)		Y	On /Of T-6, P-6 from SX	See SX	See SX	See SX	See SX	See SX
			15b	SX Inlet Flow pH		N	None	N/A	N/A	N/A	See SX	See SX
			15c		Outlet Flowrate (P-15a)	N	2 - 6 (Target 4.0 LPM)	RPM	N/A	Continuous	RPM of P-15a slaved to the Average Level Sensor in TK-15 / PLC	PLC Programming

		15d		Outlet Flowrate (P-15b)	Y*	TBD	RPM converted to flow rate via plc programming	N/A	Continuous	Set Point / PLC	Operator Adjust
		15e		pH	Y	None	N/A	N/A	N/A	N/A	N/A
		15f		Tank Temperature	Y	None	N/A	N/A	N/A	N/A	N/A
	TK-16	16a	Ascorbic Acid Solution		N	TBD	TBD on correct makeup	N/A	N/A	Makeup by operator	TBD
		16b		Outlet Flowrate (P-16)	Y	See 3b	See 3b	See 3b	See 3b	See 3b	See 3b
	TK-17	17a	Flow Rate of NaOH		N	N/A	N/A	N/A	N/A	N/A	Operator Control
		17b		Outlet Flowrate (P-17)	Y	See 4d	See 4d	See 4d	See 4d	See 4d	See 4d
	TK-18	18a	Filter Aid Make up Feed		N	TBD	N/A	N/A	N/A	N/A	
		18b		Outlet Flowrate (P-18)	N	TBD	N/A	N/A	N/A	N/A	
Water Treatment	TK-21	21a	Ca(OH)2 Acid Solution		N	TBD					
		21b		Outlet Flowrate (P-21)	Y						

						Y/N						
		TK-19	19a	Inlet Flow Rate TK-15 (P-15b)		Y*	See 15d	See 15d	See 15d	See 15d	See 15d	See 15d
			19b	Inlet Flow Temperature		N	None	N/A	N/A	N/A	N/A	N/A
			19c	Inlet Flow Rate Ca(OH)2 (P-21)		Y	TBD	RPM	N/A	Continuous	Set Point/ PLC	See 4e
			19d		pH	Y*	TBD	Digital Readout	N/A	Continuous	Set Point / PLC adjustment of 4d	Operator Adjust
			19e		Tank Temperature	Y	N/A	Thermocouple	N/A	Continuous	Display	N/A
			19f		Outlet Flowrate	N	Gravity Overflow	N/A	N/A	N/A	N/A	N/A
		TK-20	20a	Mass of Slurry		N	N/A	N/A	N/A	N/A	N/A	N/A
			20b	Inlet Temperature		N	None	N/A	N/A	N/A	N/A	N/A
			20c	Inlet Flowrate		Y	See 19f	N/A	N/A	N/A	N/A	N/A
			20d		pH	N	None	N/A	N/A	N/A	N/A	N/A
			20e		Tank Temperature	N	N/A	Thermocouple	N/A	Continuous	Display	N/A
			20f		Slurry Flowrate (P-20)	Y*	See FP2c	See FP2c	See FP2c	See FP2c	See FP2c	See FP2c

		FP - 2	FP2 a	Inlet Slurry (P-20)		Y*	TBD	?	N/A	?	?	Operator cycle
			FP2 b	Inlet Temperature		N	None	N/A	N/A	N/A	N/A	
			FP2 c	Filter Cake Solid Mass		Y	70-75%	TBD	TBD	TBD	TBD	TBD
			FP2 d	Filter Cake Liquid Mass		Y	25-30 %	TBD	TBD	TBD	TBD	TBD
			FP2 e	pH		N	None	N/A	N/A	N/A	N/A	N/A
		System Wide									
		Leach Tanks	Sys -a	Residence Time		Y	2-11 hrs	Calculation	1	Adjustment	P-13 Flow Rate	SOP
		Leach Tanks	Sys -b	Leach Liquid Recirculating Load		Y	TBD	Calculation	1	Adjustment	P-13 vs P-2 Flow Rate	SOP
				Filter Cake Washing		Y	TBD	TBD	TBD	TBD	TBD	TBD

APPENDIX 2. SAFETY PROCESS FMEA

FMEA																				

FMEA Number: 002-Hydromet Circuit Startup Safety Evaluation

FMEA Plan Owner / Phone: Josh Werner (509)995-6697

Date Original: 6/14/2018

Date Revised: 4/1/19

Process: Plant

Team: Rick Honaker, Josh Werner

Description: Operation of Pilot Plant

Approval Date: Rick Honaker 6/15/2017

Revision Note: Revised by Blanton Park on 7/31/18 and Douglas Addo to assign responsibility and target date to various FMEA actions. *Weekly JHA, where numerous operators will observe & analyze the current operating procedures with the goal of continuously working towards the safest work environments and mitigating hazards.

Process Number	Process Name/ Description	Tank, Device, Equipment	Characteristics No.	Characteristics Inputs	Characteristics Output	Potential Failure Mode	Potential Effect(s) of Failure	Severity	Potential Cause of Failures	Occurrence	Current Process Controls Prevention	Current Process Controls Detection	Detection	R.P.N.	Recommended actions	Responsibility & Target Date	Actions Taken & Completion Date	Severity	Occurrence	Detection	R.P.N.
1	Leaching	Leach Tank	1a			Leaking Acid	Acid Burn	7	Loose Fittings	7	Water Test	Visual	5	245	Water Shake Down	Joshua Werner 6/19/2018	Water Test 6/19/18	7	1	5	35
			1b			Leaking Acid	Acid Burn	7	Leaking Pipes	3	No Acids Pumped Overhead	Design	5	105	Check Off Walk Through	Jacob Gill 8/1/18					
			1c			Leaking Acid	Acid Burn	7	Acid Exposure	3	Shower/Eyewash	Design	3	63	Test Shower Montly,	Kin Craig 8/3/18					

													Test Log					
			1d		Leaking Acid	Acid Burn	7	Peristaltic Pump Wear	7	Pre and Post Shift Start Inspection Logs	SOPs	7	343	Maintenance and Replacement Schedule	Jacob Gill 8/6/18			
			1e		Contact Acid	Acid Burn	7	Inspecting Tank	7	Design	Operator	5	245	SOP and Procedure for Checking Tank	Alind Chandra 8/20/18			
			1f		Fire	Badness	10	Electric Heater	3	Low Solution Sensor	Shut Off	10	300	Test Shutoff each tank and record	Jacob Gill 6/15/18	Sensors Installed 6/19/18		0
			1g		Fire	Badness	10	Electric Induced	1	Fire Alarms	Auditory	5	50	Test Bi annually	Rick Honaker 1/1/19			
			1h		Fire	Badness	10	Electric Induced	1	Fire Extinguishers	Location	3	30	Inspect Bi annually	Rick Honaker 1/1/19			
			1i		Fire	Badness	10	Electric Induced	1	Fire Department Training	Fire Department Training	5	50	Host a Fire Department Field Trip	Joshua Werner 8/8/18			
			1j		Fall	Injury	10	Reaching, Awkward Position	5	Mobile Platform Ladder	Visualy see if operator is utilizing	3	150	JHA Observation and Improvement	*Weekly JHA			

	Component	Item			Failure Mode	Effect	SEV	Cause	OCC	Control	Detection	DET	RPN	Recommended Action	Responsibility	Action Taken	SEV	OCC	DET	RPN
		1k			Acid Fumes	Respiratory Distress	5	Poor Ventilation	5	Ventilation Design	Operator	7	175	Vent Survey	Blanton P/Jacob G 8/6/18					
		1l			Overheating	Melting Tank	10	Strong Acid Addition	7	Design	None (Will be added after water test)	10	700	JHA Observation and Improvement on Startup, Thermocouple and display	Doug Addo 8/10/18					
		1m			Leach Tank Tip Over	Acid Burns	10	Earth Quake	1	Stands and Bolt to the Floor	3	10	100		Jacob Gill 6/18/18	Stands Bolted 6/18/18	10	1	10	100
		1n			Leach Tank Tip Over	Acid Burns	10	Fork Lift Strike	3	Operator	10	10	300	Need A-Safe Barriers Installed on Acid Startup	Blanton Park 6/7/18					
		1o			Tank Failure	Acid Burns	10	See1l, 1n	3	Sump	Sump Pumps	3	90	Sump Pump Tests	Jacob Gill 6/6/18					
		1p			Tank Overflow	Acid Burn	10	Plugging outlet Line	3	Outlet Diameter	Visual	7	210	Water Only Solids Test	Rick Honaker 8/27/18					
		1q			Tank Overflow	Acid Burn	10	GFCI trips some pumps but not all	10	Wiring Design	Visual	7	700	Ensure Pumps on One Circuit/Add Level alarms	Doug Addo 8/17/18					
	Thickener Tank	1r			Leaking Acid See 1a-d															

			ID			Failure Mode	Effect	S	Cause	O	Controls	Detection	D	RPN	Recommended Action	Responsibility	Action Taken				
			1s			Acid Spray	Acid Burn	7	Unplugging a Line	5	Pump Selection	Flow Meter	5	175	Water Only Solids Test, SOP for plugged lines	Jacob Gill 8/10/18					
			1t			Fall See 1j															
			1u			Plugged Peristaltic Pump	Contact with Acid	10	Poor Pump Design	5	Pump Selection	Flow Meter	5	250	SOP and JHA for Clearing a Plug	Jacob Gill 8/10/18					
	Chemical Make Up Tank		1v			Acid Splash	Acid Burn	7	Pouring Strong Acids	7	PPE	No detection for when a splash will occur	7	343	JHA Observation and Improvement, Seek to Eliminate Hazard	*Weekly JHA					
			1w			Lime Dust	Caustic Mucus Membrane Burn	10	Pouring and Creating Dust	7	None	No detection for when a splash will occur	7	490	Eliminate with Method or Ventilation	Alind Chandra 7/25/18	Lime dust hazard eliminated via change of method (NaOH solution instead)	10	0	7	0

			1x			Overheating	Melting Tank	10	Mixing Strong Acids	7	None	None	10	700	JHA Observation and Improvement, Seek to Eliminate Hazard, Displays with Thermocouples	*Weekly JHA; Doug Addo 8/17/18				
			1y			Overheating	Melting Tank	10	Mixing Strong Bases	7	None	None	10	700	JHA Observation and Improvement, Seek to Eliminate Hazard, Displays with Thermocouples	*Weekly JHA; Doug Addo 8/17/19				
2	Solvent Extraction		2a			Fume Exposure	Health	7	Poor Ventilation	5	Design of Ventilation System	None	10	350	Ventilation Survey	Blanton P/Jacob G 8/6/18				
			2b			Fire See 1i														
			2c			Fire	Badness	10	Fire Spread from Kerosene Floating on	3	Foam Fire Extinguishers	Visual	3	90	Verify Location and Placement	Blanton Park 8/3/18				

						Water												
			2 d		Leaking Acid See 1-4													
			2 f		Acid Fumes See 11													
			2 h		Acid Contact	Acid Burn	7	Poor Ergonomics	5	Design	Design	5	1 7 5	JHA Observation and Improvement	*Weekly JHA			
			2 i		Overheating	Melting Tank	1 0	Strong Acid Addition	7	None	None	1 0	7 0 0	JHA Observation and Improvement, Seek to Eliminate Hazard	*Weekly JHA; Alind Chandra 8/10/18			
3	Refining	Solvent Extraction	3 a		See Solvent Extraction													
		Strong Acid	3 b		Operator Exposure	Acid Burn	1 0	Poor Circuit Design	5	Drip Tray	Visual	3	1 5 0	JHA Observation and Improvement, Seek to Eliminate Hazard To Operator	*Weekly JHA, Alind Chandra 8/17/18			

#	Process	Component	ID			Failure Mode	Effect	Sev	Cause	Occ	Controls	Detection	Det	RPN	Recommended Action	Responsibility	Action Taken	Sev	Occ	Det	RPN
		Strong Bases	3c			Operator Exposure	Acid Burn	10	Poor Circuit Design	5	Drip Tray	Visual	3	150	JHA Observation and Improvement, Seek to Eliminate Hazard To Operator	*Weekly JHA, Alind Chandra 8/17/18					
		Chemical Make up	3d			Operator Exposure	Acid Burn	10	Poor Process Design	5	Standard Lab Practices	Visual	3	150	JHA Observation and Improvement, Seek to Eliminate Hazard To Operator	*Weekly JHA, Alind Chandra 8/17/18					
		Fumes	3e			Operator Exposure	Respiratory Distress	5	Poor Ventilation	5	Ventilation Design	Operator	7	175	Vent Survey	Blanton P/Jacob G 8/6/18					
		Furnaces	3f			Contact Hot Piece	Burn	7	Not Using PPE	3	PPE	Visual	3	63	PPE, JHA review	Wencai Zhang 8/24/18					
4	Water Treatment	Sumps				Sump Overflow	Chemical Exposure	7	Sump Pump Failure	3	Additional berms Designed to single largest tank	Visual	3	63	Install berm systems	Blanton Park 6/19/18	Berms designed for largest tank installed 6/19/18	7	1	3	21
						Mulitiple Tank Overflow	Chemical Exposure	10	Natural Disasters	1	None	None	10	100	Evaquation / Seek Shelter Plan	Joshua Werner 8/10/18					

						Neutralization Tank Overflow	Water in Sump	5	Incorrect Control	3	Design	Visual	3	45	Overflow Testing	Jacob Gill 8/7/18					
		Tanks																			
5	Chemical Storage	Strong Acids				Mix with Bases	Spontaneous Combustion	10	Improper Storage	3	Label and Storage	Visual	3	90	Proper chemical storage areas designated with labels on containers	Blanton Park 8/10/18					
						Leak	Exposure	7	Improper Storage	3	Secondary Containment	Visual	3	63	Install secondary containment for acids and bases	Blanton Park 8/9/18					
		Strong Bases				Mix with Acids	Spontaneous Combustion	10	Improper Storage	3	Label and Storage	Visual	3	90	Proper chemical storage areas designated with labels on containers	Blanton Park 8/10/18					
						Leak	Exposure	7	Improper Storage	3	Secondary Containment	Visual	3	63	Install secondary containment for acids and bases	Blanton Park 8/9/18					

6	Material Handeling	Forklift				Strike Operator	Injury	10	Operator Error	3	Training	High Vis	3	90	Proper task training	Forklift Operator, Continuous					
						Strike Building	Damage	7	Operator Error	3	Training	Visual	3	63	Need to add Barriers	Blanton Park 8/10/18					
						Strike Equipment	Damage	7	Operator Error	3	Training	Visual	3	63	Proper task training, analyze work area prior to operation	Forklift Operator, Continuous					
						Drop Load	Injury, Damage	10	Operator Error	3	Training	Visual	3	90	Plan lifting procedure prior to operation	Forklift Operator, Continuous					
		Barrel Lifter				Pinch Points	Injury	7	Operator Error	4	Training	Visual	4	112	Proper task training, visualize location of pinch points	Barrel Lift Operator, Continuous					
						Drop Load	Injury	10	Operator Error	5	Training	Visual	5	250	Proper task training, ensure barrel straps are securely fastened prior to lifting	Barrel Lift Operator, Continuous					

7	Worksp ace						Conge stion	Slip, Trip, Fall, Struck	7	Not Using Good House keepin g Practic es	3	Traini ng	Visual	6	1 2 6	Daily inspectio n of work areas	Operato r, Continu ous				
							Visitor s	Injury	1 0	Unawa re of Hazard s	3	None	None	1 0	3 0 0	Need a Visitor Policy	Blanton Park 8/8/18				
							Electri cal Hazard s	Injury	7	Insulat or Failure	5	Prope r wiring and use of extens ion cords	Visual	3	1 0 5	Inspect extensio n cords prior to use	Operato r, Continu ous				
							Electri cal Hazard s	Injury	7	Insulat or Failure	5	No Extens ion Cords for Perma nent Fixtur es	Visual	3	1 0 5		Operato r, Continu ous				
							Electri cal Hazard s	Injury	7	Insulat or Failure	5	Forklif t Ramps	Visual	3	1 0 5	Inspect cables within forklift ramps weekly to ensure safe working order	Forklift Operato r, Continu ous				

8	Workforce					Electrical Hazards	Injury	7	Liquid	5	GFCI For 110 Power	Operator	3	105	Ensure water handling is kept away from any electrical fixtures	Operator, Continuous					
						Not Knowing Site Hazards	Injury	7	Improper Training	1	Training Records	Training Records	3	21	Site specific training for any new worker or visitor	Alind Chandra, Continuous					
						Not Wearing PPE	Injury	7	Improper Training	1	Training Records	Training Records	3	21	Signs, Proceedures	Doug Addo	Signs installed 7/23/18	7	1	3	21
						Not Trained On Tasks	Injury	7	Improper Training	1	Training Records	Training Records	3	21	Task training on any task operator has not previously performed or been trained on	Rick Honaker, Continuous					
						Not Chemical Trained	Injury	7	Improper Training	1	Training Records	Training Records	3	21	Hard Hat Stickers	Alind Chandra 8/17/18					
						Catastrophic Tank Failure	Death	10	Improper Training	1	Training Records	Training Records	3	30	Need What If Drills	Joshua Werner 8/10/18					

#	Category					Hazard	Consequence	Sev	Cause	Prob	Control	Control	Det	RPN	Recommendation	Responsible / Date					
						Catastrophic Fire	Death	10	Improper Training	1	Training Records	Training Records	3	30	Need What If Drills	Joshua Werner 8/10/18					
9	Visitors					Chemical Exposure	Injury	7	No Clear Directions	7	None	None	10	490	Create a Visual Management Plan	Blanton Park 8/6/18					
						No PPE	Injury	7	No Clear Directions	7	None	None	10	490	Create a Visual Management Plan	Blanton Park 8/6/18					
						Run Over	Death	10	No Clear Directions	7	None	None	10	700	Create a Visual Management Plan	Blanton Park 8/6/18					
						Parking	Damage	7	No Clear Directions	7	None	None	10	490	Create a Visual Management Plan	Blanton Park 8/6/18					
						Deliveries	Injury	7	No Clear Directions	7	None	None	10	490	Create a Visual Management Plan	Blanton Park 8/6/18					

APPENDIX 3 LEACH TANK 6 LADDER LOGIC PROGRAMMING

RUNG 19: PID CONTROL LOGIC

LEACH TANK 6
ACID ADDITION
****
****

Leach Tank 6
Manual/Automatic
Toggle Switch*
Grn XIO- Manual Mode
Grn XIC - Auto Mode
HMI_PH_Neutral_TK6_Auto

Rung 19:
Move Function during
Manual Mode Control
Leach Circuit pump
(L/S 10)

20

```
           ]/[                                          MOV
                                                  Move
                                                  Source      HMI_LS_10_SP
                                                                      0.0
                                                  Dest   LS_10_Command_RPM
                                                                      0.0
```

**************Acid Leach Tank 6 Control Logic*************
Rung 20: Moving Average for pH Reading to have a smooth uniform control of the pH
****

Leach Tank 6
Sends the moving
average pH value to
process variable
(PV) tag in the PID
function

21

```
                                                         MOV
                                                  Move
                                                  Source   TanK6_pH_Average
                                                                  5.077334
                                                  Dest    pH_Leach_TK6_Read
                                                                  5.077334
```

**************Acid Leach Tank 6 Control Logic*************
Rung 21- 28: Moving Average Block Function using Arrays (6)
****

LEACH TANK 6 MOVING
AVERAGE BLOCK
TK6_PH_Average.DN

LEACH TANK 6 MOVING
AVERAGE BLOCK

22

```
           ]/[                                           AVE
                                                  Average File          (EN)
                                                  Array   TK6_PH_Ave_Array[0]
                                                  Dim. To Vary        0   (DN)
                                                  Dest     TanK6_pH_Average
                                                                 5.077334  (ER)
                                                  Control   TK6_PH_Average
                                                  Length               6
                                                  Position             0
```

**************Acid Leach Tank 6 Control Logic*************
Rung 22: Timing Block for moving average (13 seconds) total

TK6_Sample_Timer.DN

23

```
           ]/[                                           TON
                                                  Timer On Delay        (EN)
                                                  Timer   TK6_Sample_Timer
                                                  Preset           13000  (DN)
                                                  Accum            3042
```

24

```
        GRT                          LES                          MOV
  Greater Than (A>B)          Less Than (A<B)              Move
  Source A  TK6_Sample_Timer.ACC  Source A  TK6_Sample_Timer.ACC  Source    TK6_PH_Current
                      3042                      3042                      5.0758753
  Source B           2000        Source B           2020        Dest  TK6_PH_Ave_Array[0]
                                                                          5.0785003
```

25

```
        ┌─────────GRT─────────┐      ┌─────────LES─────────┐      ┌─────────MOV─────────┐
        │ Greater Than (A>B)   │      │ Less Than (A<B)      │      │ Move                 │
        │ Source A  TK6 Sample Timer.ACC│   │ Source A  TK6 Sample Timer.ACC│  │ Source    TK6 PH Current│
        │                  3042│      │                  3042│      │              5.0758753│
        │ Source B         4000│      │ Source B         4020│      │ Dest  TK6 PH Ave Array[1]│
        └──────────────────────┘      └──────────────────────┘      │              5.0785003│
                                                                     └──────────────────────┘
```

26

```
        ┌─────────GRT─────────┐      ┌─────────LES─────────┐      ┌─────────MOV─────────┐
        │ Greater Than (A>B)   │      │ Less Than (A<B)      │      │ Move                 │
        │ Source A  TK6 Sample Timer.ACC│   │ Source A  TK6 Sample Timer.ACC│  │ Source    TK6 PH Current│
        │                  3042│      │                  3042│      │              5.0758753│
        │ Source B         6000│      │ Source B         6020│      │ Dest  TK6 PH Ave Array[2]│
        └──────────────────────┘      └──────────────────────┘      │              5.0758753│
                                                                     └──────────────────────┘
```

27

```
        ┌─────────GRT─────────┐      ┌─────────LES─────────┐      ┌─────────MOV─────────┐
        │ Greater Than (A>B)   │      │ Less Than (A<B)      │      │ Move                 │
        │ Source A  TK6 Sample Timer.ACC│   │ Source A  TK6 Sample Timer.ACC│  │ Source    TK6 PH Current│
        │                  3042│      │                  3042│      │              5.0758753│
        │ Source B         8000│      │ Source B         8020│      │ Dest  TK6 PH Ave Array[3]│
        └──────────────────────┘      └──────────────────────┘      │              5.0776253│
                                                                     └──────────────────────┘
```

28

```
        ┌─────────GRT─────────┐      ┌─────────LES─────────┐      ┌─────────MOV─────────┐
        │ Greater Than (A>B)   │      │ Less Than (A<B)      │      │ Move                 │
        │ Source A  TK6 Sample Timer.ACC│   │ Source A  TK6 Sample Timer.ACC│  │ Source    TK6 PH Current│
        │                  3042│      │                  3042│      │              5.0758753│
        │ Source B        10000│      │ Source B        10020│      │ Dest  TK6 PH Ave Array[4]│
        └──────────────────────┘      └──────────────────────┘      │              5.0785003│
                                                                     └──────────────────────┘
```

29

```
        ┌─────────GRT─────────┐      ┌─────────LES─────────┐      ┌─────────MOV─────────┐
        │ Greater Than (A>B)   │      │ Less Than (A<B)      │      │ Move                 │
        │ Source A  TK6 Sample Timer.ACC│   │ Source A  TK6 Sample Timer.ACC│  │ Source    TK6 PH Current│
        │                  3042│      │                  3042│      │              5.0758753│
        │ Source B        12000│      │ Source B        12020│      │ Dest  TK6 PH Ave Array[5]│
        └──────────────────────┘      └──────────────────────┘      │              5.0750003│
                                                                     └──────────────────────┘
```

Leach TK 6
PID

30

Leach Tank 6
Manual/Automatic
Toggle Switch*
Grn XIO- Manual Mode
Grn XIC - Auto Mode
HMI_PH_Neutral_TK6_Auto

The function
controls the PID
loop for pH control
in Leaching Tank 6

```
        ┌─────────MOV─────────┐
        │ Move                 │
        │ Source  HMI PH Neutral TK6 SP│
        │                  2.1 │
        │ Dest   PH_Control_Leach_TK6.SP│
        │                  2.1 │
        └──────────────────────┘
```

The function
controls the PID
loop for pH control
in Leaching Tank 6

```
        ┌─────────PID─────────┐
        │ Proportional Integral Derivative│
        │ PID          PH Control Leach TK6 [...]│
        │ Process Variable   pH Leach TK6 Read│
        │ Tieback                     0│
        │ Control Variable   LS 10 Command RPM│
        │ PID Master Loop             0│
        │ Inhold Bit                  0│
        │ Inhold Value                0│
        │ Setpoint                  2.1│
        │ Process Variable    2.0895002│
        │ Output %                  0.0│
        └──────────────────────┘
```

Leach TK 6
PID contd....

31

Leach Tank 6
Manual/Automatic
Toggle Switch*
Grn XIO- Manual Mode
Grn XIC - Auto Mode
HMI_PH_Neutral_TK6_Auto

The final average of
the arrays collected
for 6 units

```
        ┌─────────GRT─────────┐
        │ Greater Than (A>B)   │
        │ Source A    TanK6 pH Average│
        │                 5.077771│
        │ Source B  HMI_PH_Neutral_TK6_SP│
        │                  2.1 │
        └──────────────────────┘
```

Turns Pump ON/OFF
LS_10_PID_Run

APPENDIX LADDER LOGIC PROGRAMMING NEUTRALIZATION TANK 4

PID CONTROL
LEACH TANK 4 (pH NEUTRALIZATION)
****

0   HMI_PH_Neutralization_TK4_Auto
        ─┤/├─

Manual Control of
NaOH Pump to
increase pH
┌─MOV──────────────────────┐
│ Move   │
│ Source   HMI LS 3 SP │
│   50.0 │
│ Dest   LS 3 Commanded RPM│
│   50.0 │
└──────────────────────────┘

***************Neutralization Leach Tank 4 Control Logic**************
Rung 1: Moving Average for pH Reading to have a smooth uniform control of the pH reading
****

Sends the moving
average pH value to
process variable
(PV) tag in the PID
function
1
┌─MOV──────────────────────┐
│ Move   │
│ Source   TK4 PH Ave │
│   3.5519168│
│ Dest pH Leach TK4 Read │
│   3.5519168│
└──────────────────────────┘

***************Neutralization Leach Tank 4 Control Logic**************
Rung 2- 9: Moving Average function using Arrays (6)
****

2   TK4_PH_Average.DN
        ─┤/├─

┌─AVE──────────────────────────────┐
│ Average File   ──(EN)──
│ Array TK4_PH Ave Array[0]   │
│ Dim. To Vary   0   ──(DN)──
│ Dest   TK4 PH Ave   │
│   3.5519168   ──(ER)──
│ Control   TK4 PH Average   │
│ Length   6   │
│ Position   5   │
└──────────────────────────────────┘

***************Neutralization Leach Tank 4 Control Logic**************
Rung 3: Timing Block for moving average (13 seconds) total

3   TK4_Sample_Timer.DN
        ─┤/├─

┌─TON──────────────────────────────┐
│ Timer On Delay   ──(EN)──
│ Timer TK4 Sample Timer   │
│ Preset   13000   ──(DN)──
│ Accum   3225   │
└──────────────────────────────────┘

4
┌─GRT──────────────────────┐ ┌─LES──────────────────────┐ ┌─MOV──────────────────────┐
│ Greater Than (A>B)   │ │ Less Than (A<B)   │ │ Move   │
│ Source A TK4 Sample Timer.ACC│ Source A TK4 Sample Timer.ACC│ Source   TK4 PH Current│
│   3225 │ │   3225 │ │   3.808875 │
│ Source B   2000 │ │ Source B   2020 │ │ Dest TK4 PH Ave Array[0] │
│   │ │   │ │   3.808   │
└──────────────────────────┘ └──────────────────────────┘ └──────────────────────────┘

5
┌─GRT──────────────────────┐ ┌─LES──────────────────────┐ ┌─MOV──────────────────────┐
│ Greater Than (A>B)   │ │ Less Than (A<B)   │ │ Move   │
│ Source A TK4 Sample Timer.ACC│ Source A TK4 Sample Timer.ACC│ Source   TK4 PH Current│
│   3225 │ │   3225 │ │   3.808875 │
│ Source B   4000 │ │ Source B   4020 │ │ Dest TK4 PH Ave Array[1] │
│   │ │   │ │   3.810625 │
└──────────────────────────┘ └──────────────────────────┘ └──────────────────────────┘

6

```
          ┌─────GRT─────────────────────┐   ┌─────LES─────────────────────┐   ┌─────MOV─────────────────────┐
          │ Greater Than (A>B)          │   │ Less Than (A<B)             │   │ Move                        │
          │ Source A  TK4 Sample Timer.ACC│   │ Source A  TK4 Sample Timer.ACC│   │ Source      TK4 PH Current  │
          │                    3225      │   │                    3225     │   │                  3.808875   │
          │ Source B           6000      │   │ Source B           6020     │   │ Dest  TK4 PH Ave Array[2]   │
          │                             │   │                             │   │                  3.808       │
          └─────────────────────────────┘   └─────────────────────────────┘   └─────────────────────────────┘
```

7

```
          ┌─────GRT─────────────────────┐   ┌─────LES─────────────────────┐   ┌─────MOV─────────────────────┐
          │ Greater Than (A>B)          │   │ Less Than (A<B)             │   │ Move                        │
          │ Source A  TK4_Sample_Timer.ACC│   │ Source A  TK4_Sample_Timer.ACC│   │ Source      TK4_PH_Current  │
          │                    3225      │   │                    3225     │   │                  3.808875   │
          │ Source B           8020      │   │ Source B           8020     │   │ Dest  TK4 PH Ave Array[3]   │
          │                             │   │                             │   │                  2.2662501   │
          └─────────────────────────────┘   └─────────────────────────────┘   └─────────────────────────────┘
```

8

```
          ┌─────GRT─────────────────────┐   ┌─────LES─────────────────────┐   ┌─────MOV─────────────────────┐
          │ Greater Than (A>B)          │   │ Less Than (A<B)             │   │ Move                        │
          │ Source A  TK4_Sample_Timer.ACC│   │ Source A  TK4_Sample_Timer.ACC│   │ Source      TK4_PH_Current  │
          │                    3225      │   │                    3225     │   │                  3.808875   │
          │ Source B           10000     │   │ Source B           10020    │   │ Dest  TK4 PH Ave Array[4]   │
          │                             │   │                             │   │                  3.807125   │
          └─────────────────────────────┘   └─────────────────────────────┘   └─────────────────────────────┘
```

9

```
          ┌─────GRT─────────────────────┐   ┌─────LES─────────────────────┐   ┌─────MOV─────────────────────┐
          │ Greater Than (A>B)          │   │ Less Than (A<B)             │   │ Move                        │
          │ Source A  TK4 Sample Timer.ACC│   │ Source A  TK4 Sample Timer.ACC│   │ Source      TK4 PH Current  │
          │                    3225      │   │                    3225     │   │                  3.808875   │
          │ Source B           12000     │   │ Source B           12020    │   │ Dest  TK4_PH_Ave_Array[5]   │
          │                             │   │                             │   │                  3.8115      │
          └─────────────────────────────┘   └─────────────────────────────┘   └─────────────────────────────┘
```

Base PID

10

```
 HMI_PH_Neutralization_TK4_Auto                                ┌─────MOV─────────────────────────────┐
 ─┤ ├─                                                         │ Move                                │
                                                               │ Source  HMI PH Neutralization TK4 SP │
                                                               │                          2.7         │
                                                               │ Dest        PH Neutralization TK4.SP │
                                                               │                          2.7         │
                                                               └─────────────────────────────────────┘

                                                               ┌─────PID─────────────────────────────┐
                                                               │ Proportional Integral Derivative    │
                                                               │ PID          PH Neutralization TK4  …│
                                                               │ Process Variable  pH Leach TK4 Read │
                                                               │ Tieback                     0       │
                                                               │ Control Variable     LS 3 Out RPM   │
                                                               │ PID Master Loop             0       │
                                                               │ Inhold Bit                  0       │
                                                               │ Inhold Value                0       │
                                                               │ Setpoint                    2.7     │
                                                               │ Process Variable     1.5920626      │
                                                               │ Output %                  100.0     │
                                                               └─────────────────────────────────────┘
```

Base PID contd....
Condition to RUN
pump if PV < SP
STOP if PV > SP

11

```
 HMI_PH_Neutralization_TK4_Auto   ┌─────LES─────────────────────┐                    LS_3_PID_Run
 ─┤ ├──────────────────────────── │ Less Than (A<B)             │ ──────────────────────( )──────
                                  │ Source A          TK4_PH_Ave│
                                  │                   3.5519168 │
                                  │ Source B  HMI PH Neutralization TK4 SP│
                                  │                   2.7       │
                                  └─────────────────────────────┘
```

***************Acidification Leach Tank 4 Control Logic***************
PID CONTROL
LEACH TANK 4 (pH Acid Addition)

Manual/Automatic
Toggle Switch*
Grn XIO- Manual Mode
Grn XIC - Auto Mode

COMMANDED RPM FOR
PUMP

12

```
 HMI_PH_ACID_TK4_Auto                            ┌─────MOV─────────────────────┐
 ─┤ / ├────────────────────────────────────────  │ Move                        │
                                                 │ Source         HMI_LS_1_SP  │
                                                 │                    600.0    │
                                                 │ Dest  LS_1_COMMANDED_RPM1   │
                                                 │                    600.0    │
                                                 └─────────────────────────────┘
```

Acid PID

Rung 13

Manual/Automatic
Toggle Switch*
Grn XIO- Manual Mode
Grn XIC - Auto Mode
HMI_PH_ACID_TK4_Auto

Addition Block to
Add a deadband
margin to pH
setpoint before
activating the aid
PID Control Function

```
---------------ADD---------------
Add
Source A    HMI_PH_Neutralization_TK4_SP
                                      2.7
Source B                              0.1

Dest                       PH_Acid_TK4.SP
                                      2.8
```

PID Control Function
****Automatic****
Acid(H2SO4) Addition
Pump Control

```
-------------------PID-------------------
Proportional Integral Derivative
PID                        PH_Acid_TK4  ...
Process Variable      pH_Leach_TK4_Read
Tieback                                 0
Control Variable   LS_1_COMMANDED_RPM1
PID Master Loop                         0
Inhold Bit                              0
Inhold Value                            0
Setpoint                              2.8
Process Variable                1.5920626
Output %                              0.0
```

Acid PID contd.....

Rung 14

Manual/Automatic
Toggle Switch*
Grn XIO- Manual Mode
Grn XIC - Auto Mode
HMI_PH_ACID_TK4_Auto

Condition to RUN
pump if PV < SP
STOP if PV > SP

```
---------------GRT---------------
Greater Than (A>B)
Source A           TK4_PH_Ave
                    3.5519168
Source B    PH_Acid_TK4.SP
                          2.8
```

Turns Pump ON/OFF
after running the
comaprison
TK4_ACID_Pump_Run

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