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Asset Management Model for an Industrial Process Automations System in the Chemical Engineering Unit Operations Laboratory at Rose-Hulman Institute of Technology

Megan Elaine Liebman
liebmame@rose-hulman.edu

Asset Management Model for an Industrial Process Automations System in the Chemical Engineering Unit Operations Laboratory at Rose-Hulman Institute of Technology

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by

Megan Elaine Liebman

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Final Examination Report

Megan E. Liebman
Name

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EXAMINATION COMMITTEE:

	Thesis Advisory Committee	Department
Thesis Advisor:	Atanas Serbezov	CHE
	Daniel Anastasio	CHE
	Craig Downing	EMGT

PASSED X **FAILED** ____

ABSTRACT

Liebman, Megan Elaine

M.S.Ch.E

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Asset Management Model for an Industrial Process Automations System in the Chemical Engineering Unit Operations Laboratory at Rose-Hulman Institute of Technology

Thesis Advisor: Dr. Atanas Serbezov

In the Chemical Engineering curriculum, the Unit Operations (UO) Laboratory is recognized as the place where hands-on exposure to industrial practices occurs. Students not only examine fundamental engineering principles, but also gain knowledge of the intricacies of modern control technology. The UO Laboratory at Rose-Hulman Institute of Technology houses an industrial state-of-the-art distributed control system, which operates eleven large-scale experiments and interfaces with approximately two hundred process instruments. The size and complexity of the distributed control system and its associated equipment have grown over the past ten years and have reached a point where a systematic asset management approach is needed to continue to deliver undisrupted support to the educational process.

The developed asset management model provides a framework to efficiently operate assets and add new assets in a consistent manner. The model includes two information repositories: one

for equipment and one for procedures and documentation. The existing equipment in the UO Laboratory has been catalogued in an equipment database, which now serves as a single point information source. A new format for a standard operating procedure (SOP) has been developed based on best industrial practices, and the existing operating instructions for all eleven large-scale experiments have been rewritten using the new SOP layout. In addition, the technical documentation associated with the eleven large-scale experiments has been collected from multiple sources and has been organized in a single location accessible by faculty, staff, and students. Critical equipment components have been identified and outside companies have been contracted to provide emergency support and scheduled maintenance. The developed asset management model has been successfully implemented in the UO Laboratory and has significantly contributed to the quality of instruction and learning.

ACKNOWLEDGEMENTS

Many thanks go to my advisor, Dr. Atanas Serbezov, for his extensive support, guidance, understanding, and encouragement throughout my studies. Thank you to the members of my committee, Dr. Daniel Anastasio and Dr. Craig Downing, for their invaluable advice and feedback.

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Most of all: Dad, Mom, and Lauren. I wouldn’t have made it here without you. Thank you for all the love, laughter, and support.

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AMS	Asset management system
DCS	Distributed control system
EIT	Enterprise Information Technology
ISO	International Organization for Standardization
RTD	Resistance temperature detector
SOP	Standard operating procedure
UO	Unit operations
VFD	Variable frequency drive

1. INTRODUCTION

Established in 1874, Rose-Hulman Institute of Technology is located in Terre Haute, Indiana. It has a rich history in chemical engineering, having awarded the United States' first degree in chemical engineering in 1889 [1]. Since then, the college has continued to offer an exceptional program to develop chemical engineers. A unique feature of the chemical engineering program at Rose-Hulman Institute of Technology is its Unit Operations Laboratory. Beginning in 1984, experiments and procedures have been added and modified to create the current laboratory experience.

The Unit Operations Laboratory is a two-story laboratory containing approximately twenty individual experiments. The main purpose of the Unit Operations Laboratory is to facilitate the laboratory project courses that students of the Chemical Engineering Department take during their junior and senior years. In these courses, the students work with five different experiments by the time they graduate and learn concepts of chemical engineering used in industry.

Eleven of the twenty experiments in the Unit Operations Laboratory utilize a process automation system, called DeltaV, which has been integrated into the Unit Operations Laboratory over the past ten years by various faculty members of the Chemical Engineering Department. The benefits of this process automations system include enabling a wider variety of experimental goals through automatic control and emulating the industrial environment with centralized data acquisition and historization. However, the integration of an industrial control system in an educational setting presents an operational and managerial challenge. It requires more complex

installation and commissioning than simple equipment would. The operations become more complex, due to a higher number of control and electronic interactions and more challenging goals. The incorporation of field instruments and computer infrastructure into the Unit Operations Laboratory requires an increased capital investment. Having more components leads to increased maintenance needs and creates additional tasks and responsibilities for the existing personnel, the faculty and the technicians of the Chemical Engineering Department. These personnel then become the bottleneck when changes to the experiments or DeltaV are proposed.

The development and implementation of an asset management model for these experiments and the DeltaV system will help alleviate personnel bottlenecks, improve the quality of instruction, and provide a common place for the accumulation of resources. The asset management model will increase the capability of the Unit Operations Laboratory to operate in an efficient manner, therefore becoming more beneficial to the students of Rose-Hulman Institute of Technology.

2. BACKGROUND AND THEORY

2.1. Defining asset management

Asset management is a systematic process of deploying, operating, maintaining, upgrading, and disposing of assets in a cost-effective manner [2]. In the chemical engineering industry, the term 'assets' most commonly refers to the operating resources of a plant, such as the process instruments and other pieces of equipment. The specific assets of the Unit Operations Laboratory will be delineated in a subsequent section.

2.2. Literature review

Asset management serves as "a tool to improve regulatory compliance; lower operation and maintenance costs; assess criticality, capacity, and life-cycle costs of key facilities; and support environmental sustainability and stewardship" [3]. As an educational establishment, the Unit Operations Laboratory does not have to follow industrial standards. However, creating a consistent structure that echoes industrial standards for the documentation of each experiment will serve to both introduce students to the concept of regulatory compliance and instill in them the value of clear and comprehensive documentation. By reducing the amount of time wasted by students or faculty searching for information that was poorly or sparsely recorded, asset management will lower the operation costs of the Unit Operations Laboratory.

Establishing a database of assets will support environmental sustainability by enabling students to access instrument information and manuals digitally, as opposed to printing them out. This database will also assist in the instruments being efficiently tracked and monitored for

maintenance needs, lowering the maintenance costs. Asset management strategies have "enabled companies to move to a predictive maintenance mode. The savings during startup alone have been $150 to $200 per device" [4]. Predictive maintenance uses the condition of in-service equipment to determine warranted maintenance tasks. When compared to routine maintenance, which is based on a time schedule, predictive maintenance saves money by reducing both the frequency that process must be halted and the length of time that the process is down for maintenance.

Some elements of asset management can be found in industries other than chemical engineering. For example, the International Organization for Standardization (ISO) provides a set of basic quality management principles, called ISO 9000, in which the companies can be certified. These principles include management responsibilities, internal audits, design control, process control, process identification and traceability, inspections and testing, document control, and quality records [5]. Because it is concerned with the process an organization uses, rather than the product or the service, ISO 9000 is applicable to many industries, including law firms, consulting engineers, and standards developers [6]. Developing an asset management model that emulates these principles as they apply to the Unit Operations Laboratory will encourage a culture of best practices.

3. ASSETS OF THE UNIT OPERATIONS LABORATORY

3.1. Description of the Unit Operations Laboratory

The Unit Operations Laboratory occupies two interconnected spaces: a two-story high bay laboratory and a single-story low bay laboratory. Each experimental unit illustrates the fundamentals of a particular unit operation or core chemical engineering concept, such as heat transfer, distillation, membrane separation, fluid flow, et cetera. Eleven of the experiments are operated with an industrial process automations system, DeltaV. DeltaV utilizes distributed control, in which each experiment is treated as a plant area and is functionally isolated from the others.

The enrollment in the laboratory project courses is usually between sixty and seventy students per term, with each term lasting for ten weeks. The students work in groups of three for six four-hour long sessions to operate a particular experiment and complete the objectives of the experimental program. To fulfill the degree requirements for a Bachelor of Science in Chemical Engineering at Rose-Hulman Institute of Technology, students must successfully complete five different experiments over three terms. A written report is submitted at the end of each experiment. Three out of the five experiments are presented to the class and faculty in a formal setting followed by a questions and answers session. The primary objectives of the laboratory project courses are related to the nature of the unit operation and typically include determination of heat or mass transfer coefficients, reaction conversion, performance parameters such as purity and recovery for separation operations, efficiency of pumps, and friction factors in piping networks.

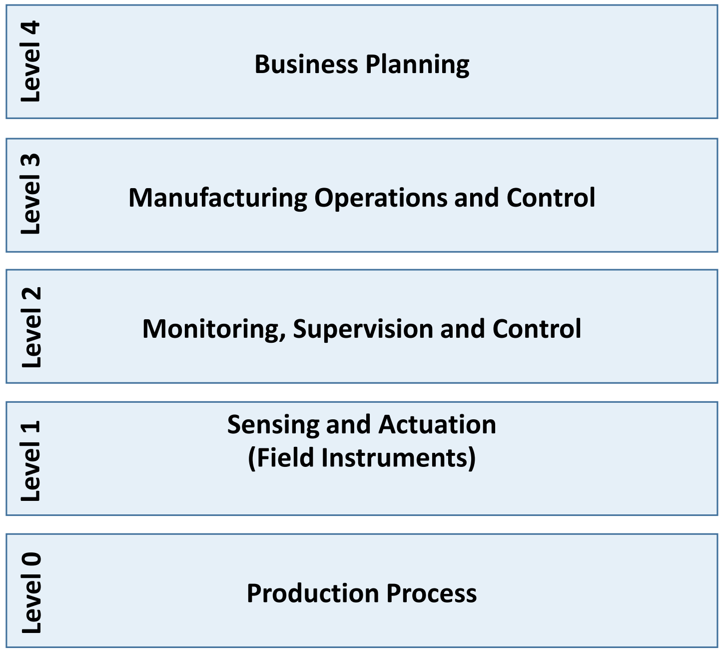

Figure 3-1: Purdue Enterprise Reference Architecture

The Purdue Enterprise Reference Architecture, shown in Figure 3-1, is a hierarchical structure of modern process manufacturing systems [7]. Each level, from the foundation up, is distinctly represented by the assets of the Unit Operations Laboratory.

3.2. <u>Process equipment and instruments</u>

Level 0 of the hierarchy is represented by process equipment, such as pumps and motors. Level 1 is represented by all the field instruments, including valves and measurement devices—sensors for level, flow, pressure, temperature, conductivity, and more. The Unit Operations Laboratory contains hundreds of process instruments. Each piece of equipment or instrument is assigned a unique device tag, which corresponds with the device type and its location. These devices form the base of the hierarchy; providing accurate and easily-accessible documentation

is critical, but the sheer number of devices means that maintaining this documentation is a challenge.

Some instruments include their own displays that allow the students to read measurements and are not connected to the control system; some have no display but send a signal to the control system; and some have both a display and a connection to the control system. This connection to the control system is accomplished via a DC signal that is transmitted from a sensor to a controller through a pair of wires. The signal ranges from 4 milliamps at zero percent of the instrument's range to 20 milliamps at one hundred percent of the range.

3.3. DeltaV process automations system

Distributed control systems allow a plant to have control elements dispersed throughout the process and connected by a communications network, rather than a having single controller at a central location. In the specific application to the Unit Operations Laboratory, the DeltaV process automations system serves as level 2 of the Purdue Enterprise Reference Architecture and uses distributed control to give each experiment its own virtual "area" – controllers and network cards that correspond to only one or two experiments. This enables work to be done on an experiment without affecting the operational capabilities of the rest of the laboratory. The designations of the experiments and areas are listed in Table 3-1, while Figure 3-2 shows how the experiments are linked to the controllers.

Table 3-1: DeltaV Experiment and Area Designations in the Unit Operations Laboratory

Area	Experiment
100	Filter Press
150	Reverse Osmosis
200	Corning Column
250	Agitated Tank
300	Liquid Flow
400	Instrumentation and Control
500	Tubular Reactor
600	Microfiltration
700	Pumps
800	Multipass Heat Exchanger
900	Fluidized Bed

Figure 3-2: DeltaV Controllers and Experiments in the Unit Operations Laboratory

The DeltaV system is hosted on several servers located in a control room within the Unit Operations Laboratory. The configuration of the servers is shown in Appendix A. One server, called the Historian, stores all the data collected by all the instruments connected to DeltaV. The Pro+ server is used to configure DeltaV and store system graphics. Each experiment that utilizes DeltaV has its own local computer. These computers use Microsoft's Remote Desktop Connection to access the Remote Server, which provides the gateway into the rest of the DeltaV system through a redundant pair of network switches.

The entire arrangement of servers and computers is typically isolated from the Rose-Hulman Institute of Technology network, with the exception of a few hours a year to update software. This was intentionally designed in order to reduce the risk of viruses and other malware being transferred via internet access.

3.4. Personnel

The personnel of the Unit Operations Laboratory include the faculty members of the Chemical Engineering Department of Rose-Hulman Institute of Technology as well as a laboratory technician. These personnel must be considered as resources, as their time is limited and valuable. Documenting the various procedures that these personnel frequently use or explain to students will eliminate unnecessary tasks and allow the personnel more time to provide educational value.

4. APPROACH AND METHODS

4.1. Creating procedures and document templates

Level 3 of the Purdue Enterprise Reference Architecture includes the operation of the experiments. DeltaV has been incorporated into the Unit Operations Laboratory in two ways: it has either been integrated into an already existing experiment, or a new experiment has been proposed that includes DeltaV elements. Without a standard documentation procedure, each faculty member has utilized their own system for recording information about the experiments that they have developed. This process has led to the existence of many inconsistent records and templates in the Unit Operations Laboratory. Additionally, students and faculty members occasionally make changes to the equipment or the procedures without notifying the department head, the laboratory technician, or other users of the experiment and without recording and documenting the change. This causes confusion when the subsequent person attempts to run the experiment, and may lead to improper techniques, damaging equipment, or even injuries.

Creating a standard template for procedures and other documents will reduce the waste of resources that occurs if the laboratory technician had to stop his work to explain procedures or share information that could not be found elsewhere and when the faculty members were not familiar with the experiment or had to consult other faculty members. Standardization will also streamline the process for students as they switch from one experiment to the next, eliminating the need for students to adapt their established experimental techniques and allowing them to devote more time and energy to learning with the equipment.

4.2. Scope for the engagement of internal and external resources

With regards to the Unit Operations Laboratory, business planning—the uppermost level of the Purdue Enterprise Reference Architecture—includes engaging both internal and external resources to provide support.

Internal resources are defined as those who are employed by Rose-Hulman Institute of Technology in various functions, such as students, technicians, faculty members, and the staff members of Rose-Hulman Institute of Technology's Enterprise Information Technology (EIT) center. External resources are not employed by Rose-Hulman Institute of Technology. The external resources that are commonly engaged for assistance with the DeltaV experiments in the Unit Operations Laboratory are Cornerstone Controls and Endress+Hauser, two companies whose products form key components of the experiments. The portion of the Unit Operations Laboratory for which each resource provides support is delineated in Appendix A.

Each internal resource serves a distinct purpose in the tasks of the Unit Operations Laboratory. Students operate the experiments, learn the principles of chemical engineering, and can be contracted to perform more in-depth research. The laboratory technician is responsible for enforcing safety procedures, maintaining the equipment in working condition, troubleshooting any malfunctioning or nonfunctioning equipment, and managing all installation, repair, and maintenance tasks. The Chemical Engineering Department faculty members initiate the process of establishing a new experiment, retain primary responsibility for the experiment throughout its usage, and supervise and support students in their tasks. The staff members of the EIT department provide assistance to the Chemical Engineering Department with the connections from the DeltaV server to the local operator stations and the Rose-Hulman Institute of Technology network.

Cornerstone Controls is a company that handles regional distribution of the DeltaV platform. For the Unit Operations Laboratory, they assist with hardware design, system architecture, licensing, and software design, development, and integration. They have been contracted to conduct quarterly audits of the DeltaV system. These audits usually consist of handling a list of changes that the faculty wish to be made, such as reconfiguring alarms.

Endress+Hauser manufactures, sells, and provides service for industrial process instruments, including dozens in the Unit Operations Laboratory. Endress+Hauser has entered into a calibration agreement with the Chemical Engineering Department, to service ten instruments per year during a one-day visit to campus. The Unit Operations Laboratory also utilizes Endress+Hauser’s equipment database, the W@M Portal, which stores device-specific information such as serial number, date of manufacture, and technical documents and manuals.

5. ASSET MANAGEMENT MODEL

5.1. Procedure for the deployment of new assets

One of the old techniques of the Unit Operations Laboratory that was detrimental to its best practices was the way that new assets were introduced into the DeltaV system. Often a faculty member or technician would upgrade or replace an instrument without notifying the other users of the experiment and without documenting the change. This typically caused confusion and wasted time when subsequent faculty members or students attempted to run the experiment for the first time, and lacked the appropriate knowledge.

A sample of this asset data entry document—for Endress+Hauser instruments—is shown in Appendix C. The document also contains guidelines for non-Endress+Hauser instruments, control valves, resistance temperature detectors (RTDs) and thermocouples, variable frequency drives (VFDs), pumps, agitators, analytical instruments, and DeltaV I/O cards. This document was divided by device type in order to streamline the process for asset data entry: if a personnel member wishes to add a new asset to the database, they must only open the document to the correct device type and then follow the indicated steps to submit all necessary information about the device.

5.2. Documentation portfolio for operation

An accurate and concise standard operating procedure (SOP) for each experiment is essential to maintaining safety and best practices in the Unit Operations Laboratory. As DeltaV had been integrated over the past eight years, several complications had developed: some information had been passed on between faculty, technician, and students only verbally, and

inconsistent documentation made it difficult for students to transition between experiments. By synthesizing all known information into a consistent template, the asset management model will help apply the ISO 9000 principles of management responsibilities, internal audits, inspections and testing, and quality records to the Unit Operations Laboratory.

Additionally, feedback from graduates of the Chemical Engineering Department was taken into account and led to the creation of a procedural checklist in addition to a standard operating procedure. The purpose of this checklist is to facilitate quicker and smoother runs of the experiment after the students are familiar with the full SOP, while still providing all necessary information and accountability. Complete SOPs and checklists for the Filter Press, Corning Column, and Tubular Reactor experiments can be found in Appendices C, D, and E, respectively.

5.3. Physical asset management

As previously mentioned, problems can arise when the correct information is not known and cannot easily be found for an instrument. Endress+Hauser's W@M Portal is an information management system that facilitates easy tracking of assets as they are added to, modified, and removed from an experiment. Figure 5-1 shows the home page of the Unit Operations Laboratory's W@M Portal. It has a brief overview of how many instruments are catalogued in the database, and the Area links on the left-hand panel provide intuitive structuring of the asset data.

5.4. Documentation management

Two repositories were created with the goal of effectively creating, storing, and distributing documents generated in association with the Unit Operations Laboratory. The first, a Microsoft SharePoint site, is a collection of the documents required for operating an experiment: DeltaV experiment-specific resources, literature resources, operational resources (SOPs and checklists), and schematics and diagrams. Documents about the DeltaV distributed control system—backups

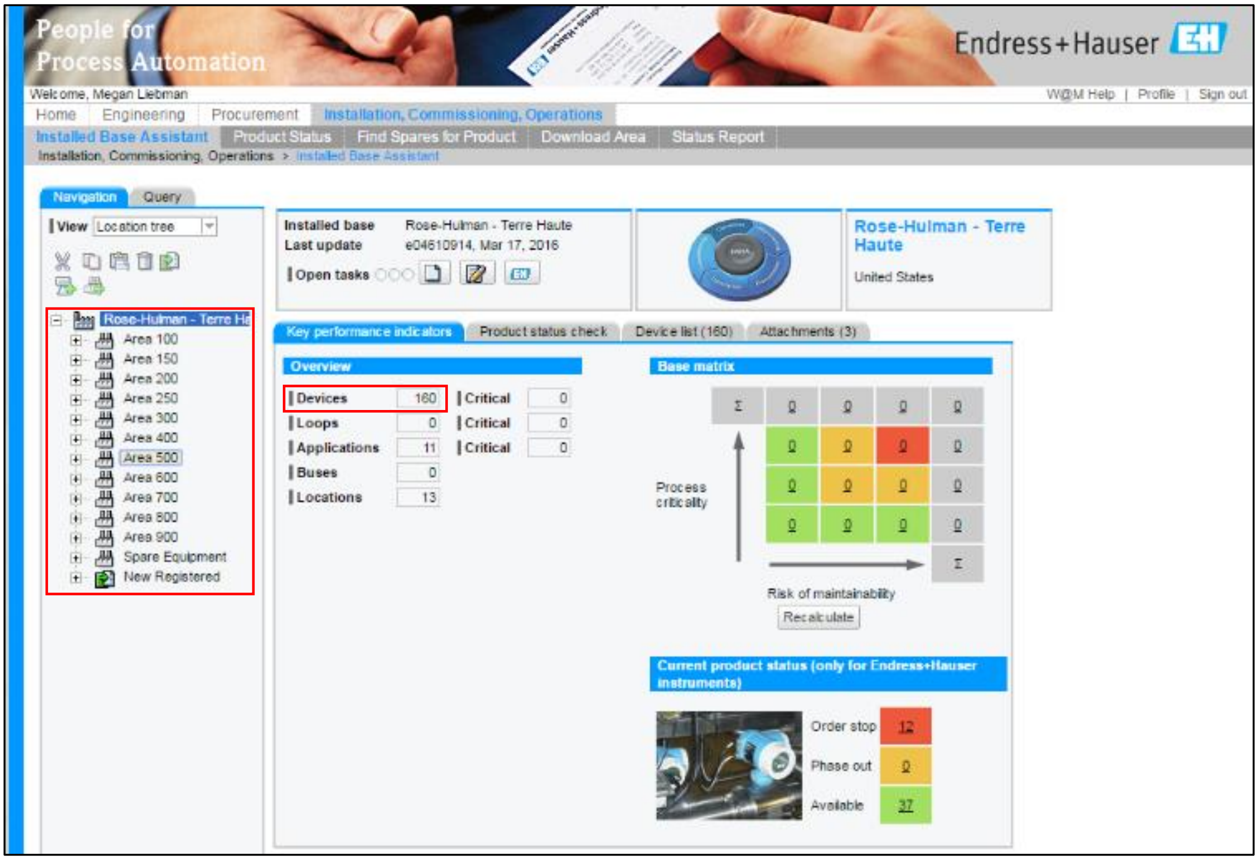

Figure 5-1: Endress+Hauser's W@M Portal

of the data historian templates and information about the network structure and the local operator stations—are also available on the SharePoint site. Permissions are tightly controlled on this site in order to maintain its organization and establish version control, ensuring that the most recent updates are accessible by all users of the experiment. Generally, faculty and staff members are allowed to read and write to the SharePoint site, while students are only allowed to read. A total of 99 documents have been uploaded.

The second repository is Endress+Hauser's W@M Portal. This is used to compile important information about the pieces of equipment as well as the technical resources associated

with each one. The information about the equipment includes its tag number, serial number, device type, manufacturer, date of manufacture, and software version; the technical resources include manuals, specifications, and calibration certificates. A total of 158 pieces of equipment have been catalogued.

In conjunction with these document repositories, a change management procedure was created, shown in Figure 5-2. The arrows indicate information flow, and the paths that a new document or a proposed change could take. Implementing this procedure ensures that the appropriate personnel are notified of a change and that it is properly recorded and documented.

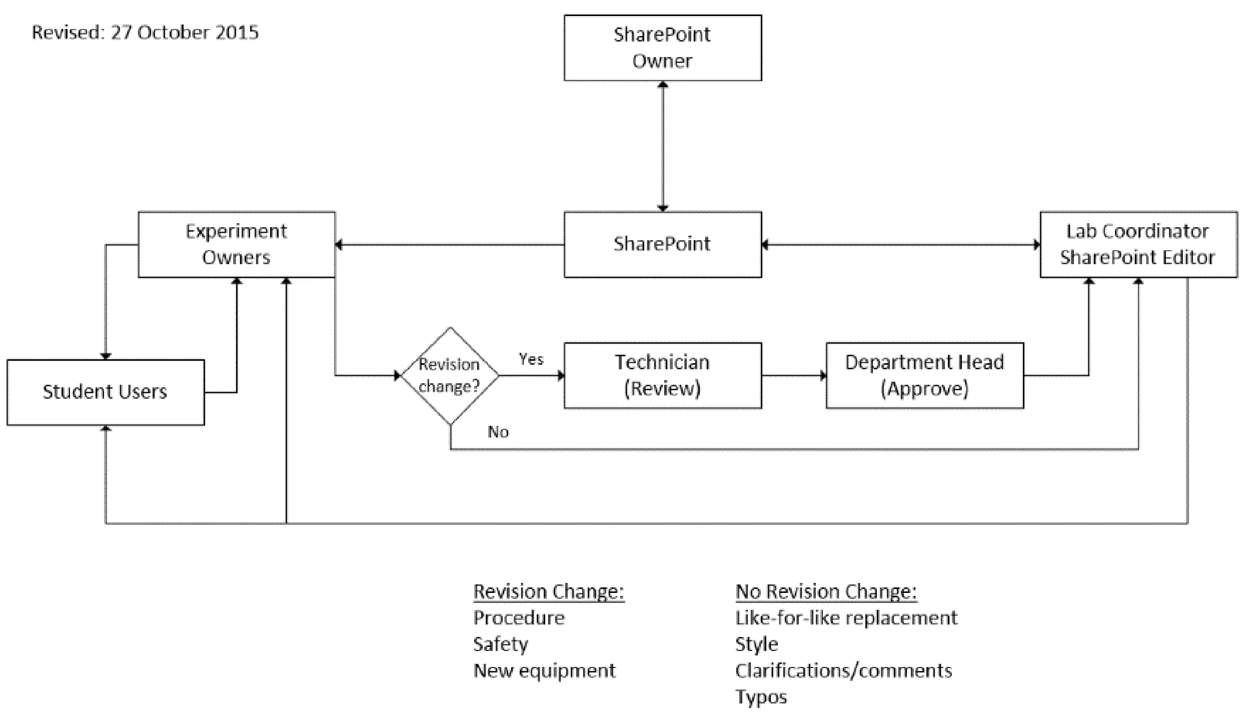

Figure 5-2: Change Management Procedure

5.5. Impact on the Unit Operations Laboratory Instruction

The asset management model was deployed at the beginning of the 2016 academic year. Four out of nine faculty members in the Unit Operations Laboratory were newly hired that year. Using the documentation in the asset management portfolio, the new faculty were able to smoothly transition into instructional roles, easily obtaining the information they needed with the resources at their disposal.

6. CONCLUSIONS

Due to the standardized templates for the documentation of experiments, equipment, and procedures, the Unit Operations Laboratory has operated more efficiently. The faculty members, the students, and the technician no longer have to go hunting for information; they know where to look and can trust that the information will be there. The creation of two information repositories has provided structure to the hundreds of documents associated with the Unit Operations Laboratory.

Additionally, the students are more prepared for industrial standards and regulations that they will encounter throughout their careers. The standard operating procedures have been updated to more accurately reflect best industrial practices; assets are tracked as they are added to, modified, and removed from experiments.

In these ways, the implementation of an asset management model in the Unit Operations Laboratory has benefited the students of Rose-Hulman Institute of Technology.

7. FUTURE WORK

In the future, updating the documentation and maintaining the version control of the databases will be critical to the success of the asset management model. Additionally, there are several areas in which the model could be expanded, including cataloging the control system components and the spare equipment in the W@M Portal and adding documentation of the control system configuration to the SharePoint site. This will provide a more comprehensive framework for the Unit Operations Laboratory.

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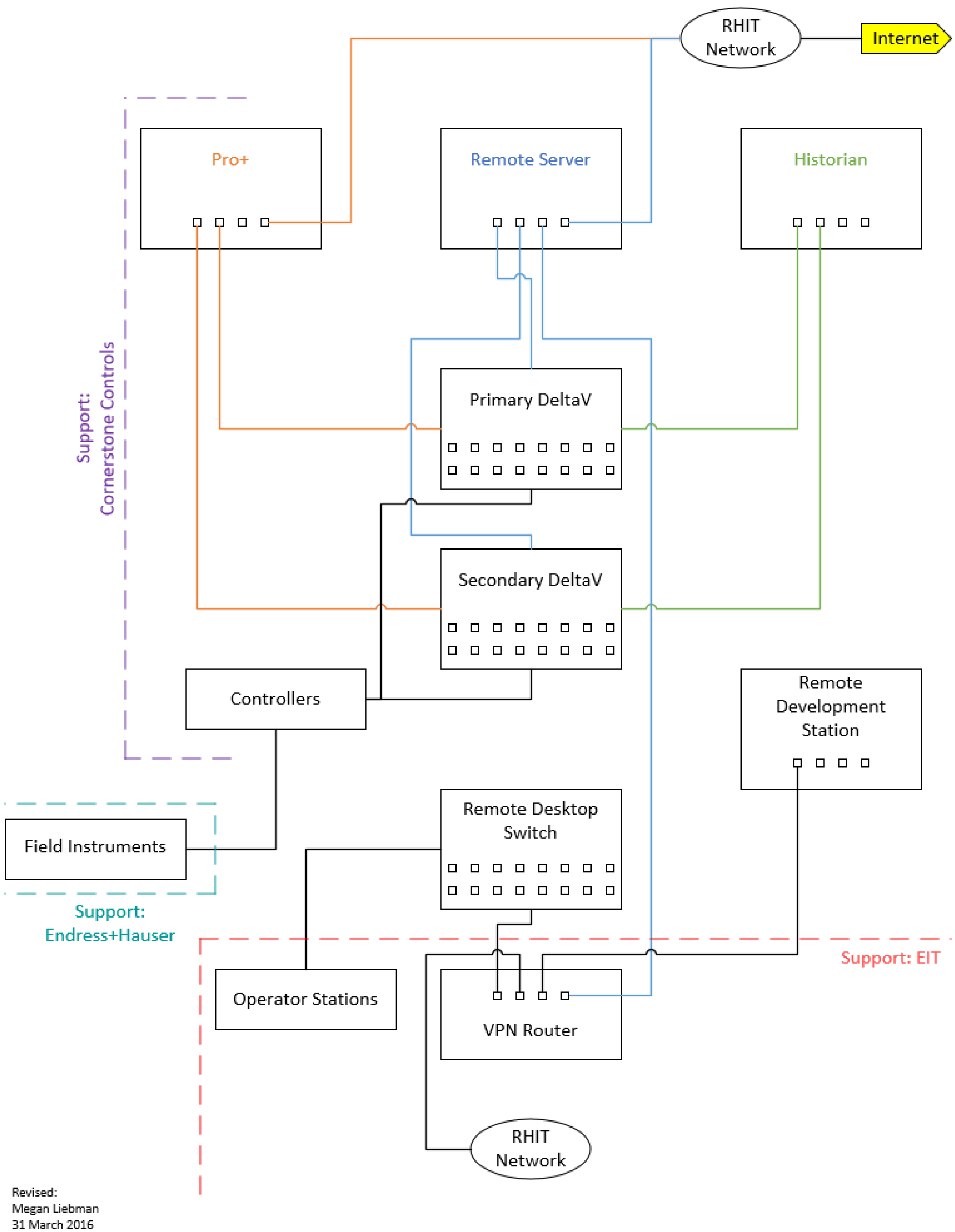
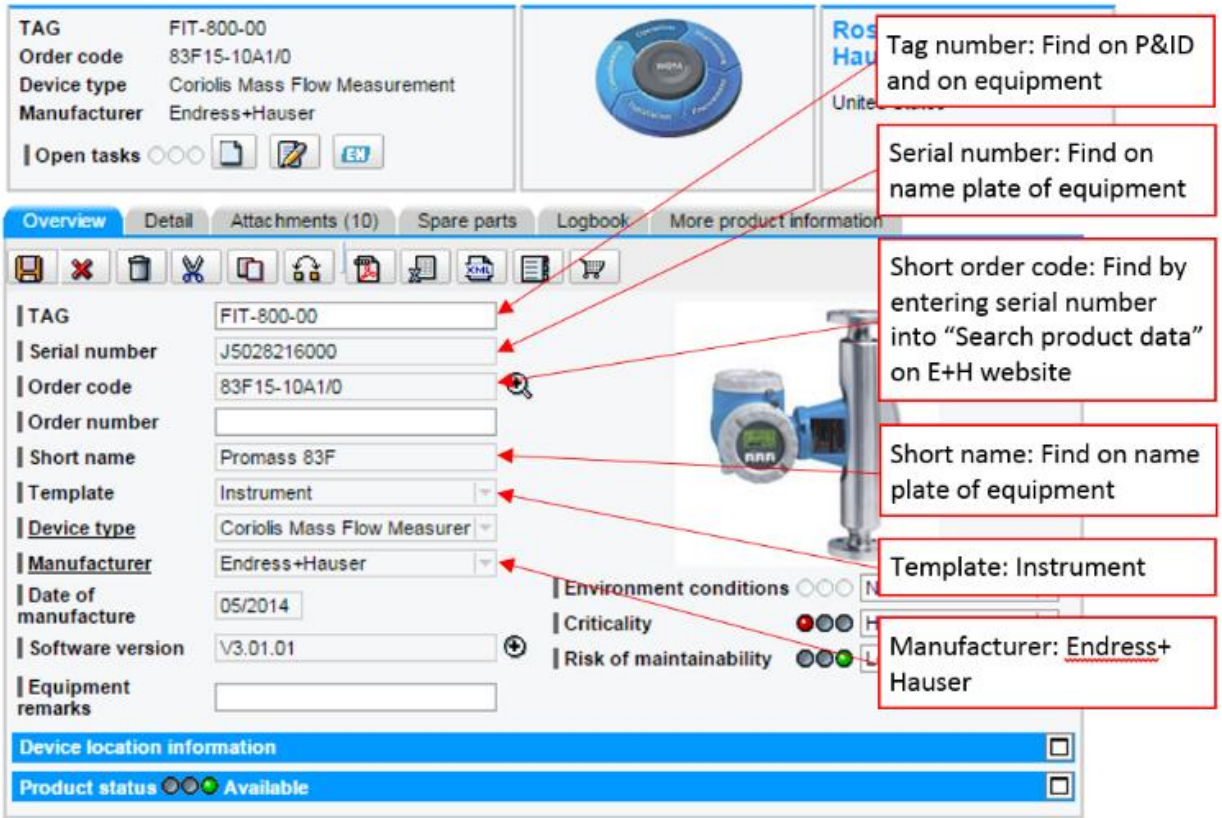
APPENDICES

APPENDIX A: DeltaV Network Structure



APPENDIX B: Guidelines for Asset Data Entry in the W@M Portal

E+H Instruments



APPENDIX C: Filter Press Standard Operating Procedure and Checklist

STANDARD OPERATING PROCEDURE DOCUMENT

Area 100 – Filter Press	Revision 1.1 Effective 8 February 2016	Page **1** of **27**

Supersedes:	Revision 1.0
Purpose:	The purpose of this document is to describe the setup and operation of the Filter Press experiment. The Filter Press experiment is used in the undergraduate Chemical Engineering Laboratory courses to study the unit operation of cake filtration. **In-Scope** This document applies to the use of the Filter Press experiment in the context of the undergraduate Chemical Engineering Laboratory courses. **Out-of-Scope** This document does not apply to uses of the Filter Press experiment outside of the undergraduate Chemical Engineering Laboratory courses.
Area(s) Involved:	Chemical Engineering Laboratory, Area 100 – Filter Press
Reasons for Revision:	Updated the location of SDS documents.

STANDARD OPERATING PROCEDURE DOCUMENT

Area 100 – Filter Press	Revision 1.1 Effective 8 February 2016	Page **2** of **27**

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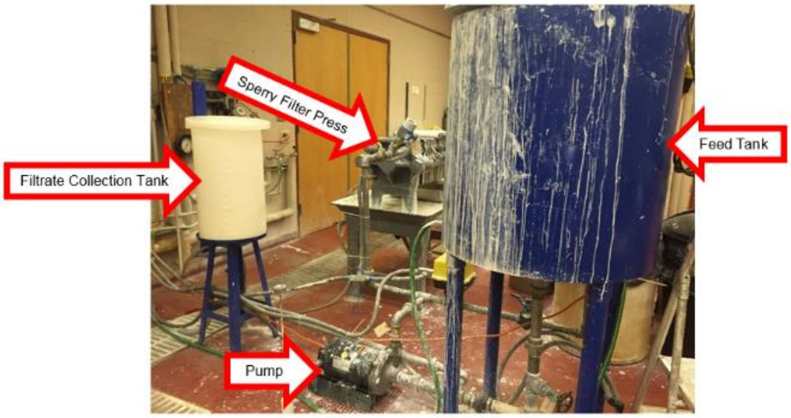
EQUIPMENT OVERVIEW

1. Experimental Setup



The Filter Press setup consists of an agitated jacketed feed tank, a pump, a Sperry plate and frame filter press, and a filtrate collection tank. The DeltaV process control and data acquisition system enables constant pressure drop operation and records all measured process variables.

Refer to the P&ID document for details.

2. Operational Ranges

Pressure Drop Across the Filter: 30 psid to 45 psid
Slurry Concentration: 10 to 12 wt% solids

SAFETY PRECAUTIONS

Kinetic Hazard: Shut down the agitator prior to opening the feed tank lid.

Pinching Hazard: Use caution when tightening or loosening the plate and frame assembly.

Chemical Hazard: Calcium carbonate can dry and crack skin. Use gloves for protection.

Chemical Hazard: Calcium carbonate particles can cause eye damage if trapped under a contact lens. Use goggles if wearing contact lenses.

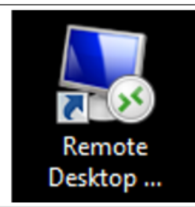
Review the SDS document for each chemical prior to conducting any experimental runs. SDS documents are located in an online database; the link is provided in the CHE Laboratory page on Moodle.

Refer to the Risk Analysis document for details.

STANDARD OPERATING PROCEDURE DOCUMENT

Area 100 – Filter Press	Revision 1.1 Effective 8 February 2016	Page **5** of **27**

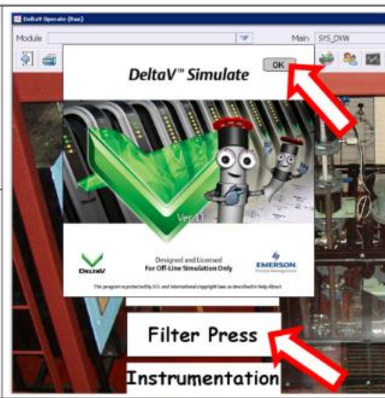
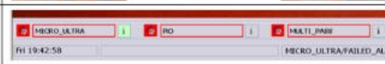
DELTAV LOGON

Step	Action and Rationale	Image
1	Turn on the Remote Operator station CHELAB-9 next to the experimental setup. Note: Normally the computer will be on, so only the monitor will need to be turned on.	
2	Log on to the computer: • Username: .\che (default) • Password: student	
3	Double-click the "Remote Desktop …" icon on the desktop.	

STANDARD OPERATING PROCEDURE DOCUMENT

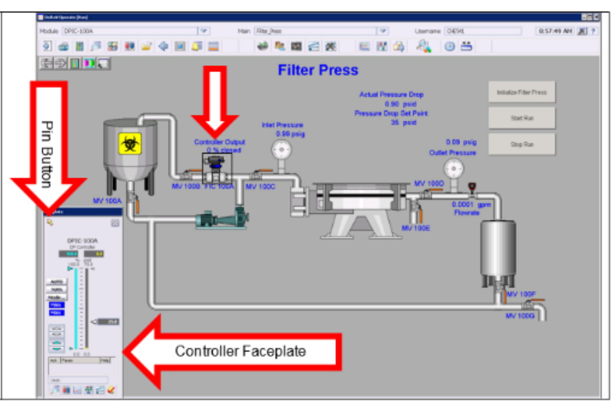
Area 100 – Filter Press	Revision 1.1 Effective 8 February 2016	Page **6** of **27**

4	Connect to the default IP address (192.168.1.201): • Username: filterpress (default) • Password: filterpress	
5	Wait for approximately 30 seconds, until DeltaV initializes.	
6	When the FlexLock ribbon appears, click on the "DeltaV Operate" button.	

7	When the DeltaV Simulate window appears, select "OK" to acknowledge the license agreement.	
8	Click on the "Filter Press" button on the DeltaV main screen.	
9	Ignore any alarms that may be flashing at the bottom of the screen.	

GRAPHICAL USER INTERFACE (GUI)

1. Experiment Main Display

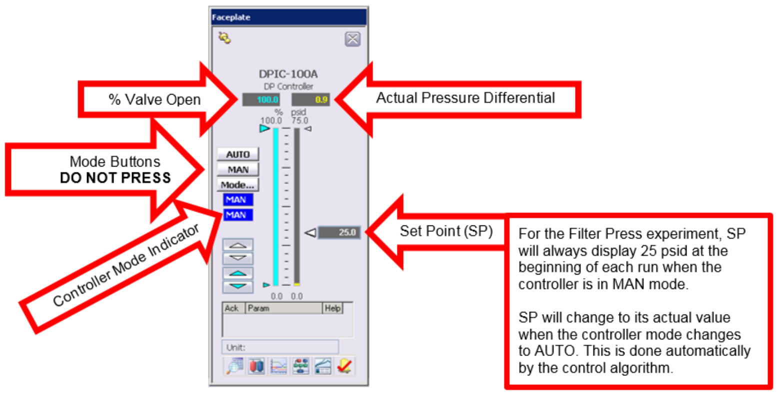
This is the GUI for the Instrumentation and Filter Press experiment. If the process variables are not displayed when you open the file (i.e., the values appear as *** or @@@), do not proceed—call the lab technician or your lab instructor. To display the controller faceplate, click on the image of the control valve (FIC-100A) and drag the faceplate to the position shown above. Pin the faceplate to the display using the Pin button.	

2. Controller Faceplate

The Controller Faceplate shows and allows access to the most important operating parameters of the control algorithm.

Important: In the Filter Press experiment the Controller Faceplate should be used to only monitor the status of the experiment. Interacting with the controller faceplate will interfere with the control algorithm and will result in a failed experiment.

The callouts in the printscreen below describe some of the most important fields in the Controller Faceplate.

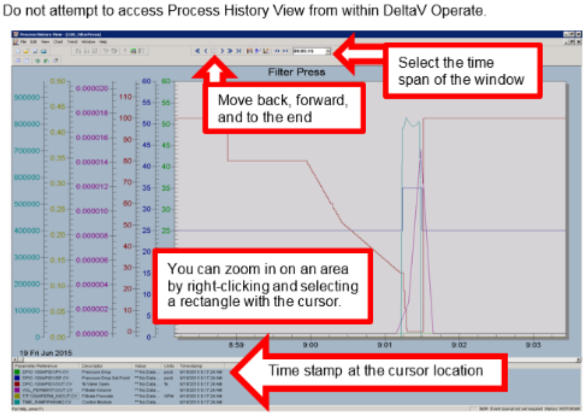


3. Process History View (PHV) Application

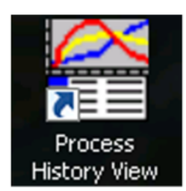
The Process History View (PHV) is an application that provides real-time and historical data in a chart recorder graphical format. It is used to monitor the progress of the experiment. It is also used to determine the start and end time in the process data retrieval procedure.

To access the Process History View utility, switch to Desktop view and double-click on the Process History View icon (1). The Process History View for the filter press will load automatically. If it does not, go to "File" and select "100_FilterPress.phvc" from the pane with recently accessed files (2).

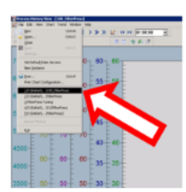
Note: Do not attempt to access Process History View from within DeltaV Operate.



(1)



(2)



The callouts in the printscreen above describe some of the most important functionalities in PHV. Clicking on the name of a variable at the bottom of the screen will bold the plotted line and make it easier to track that particular variable.

STANDARD OPERATING PROCEDURE DOCUMENT

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PROCEDURAL DESCRIPTIONS

1. Equipment Preparation

Step	Action and Rationale	Image
1	Review the appropriate SDS documents and the chemical agent release response plan for this experiment.	
2	The control valve FIC-100A must be in the fully open position. Look at the top of the valve. If the indicator is not in the fully open position, do not proceed further. Contact your instructor or Mr. Cunning.	
3	Check that these manual valves are fully OPEN: • MV-100B • MV-100D • MV-100I	
4	Check that these manual valves are fully CLOSED: • MV-100A • MV-100C • MV-100E • MV-100F • MV-100G • MV-100J	

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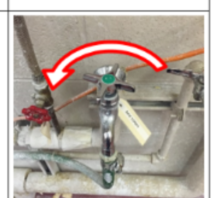
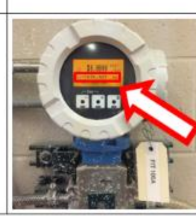
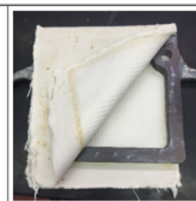
5	Check the level of water in the feed tank so that the liquid is at least 2 to 3 inches above the return pipe at the bottom of the feed tank.	
6	Break the settled chalk in the feed tank with the broom handle or the plastic paddle, making sure all the settled chalk is fully suspended.	
7	Turn on the cooling water by opening manual valve MV-100H fully to the left.	
8	Turn on the agitator in the feed tank.	

9	Open MV-100A fully to the left. Since MV-100B and FIC-100A are already open, opening MV-100A will send the feed through the pump and back into the feed tank. This will recycle the slurry mixture to achieve a uniform tank concentration.	
10	Turn on the pump using the "Pump Power" switch (on the South wall).	

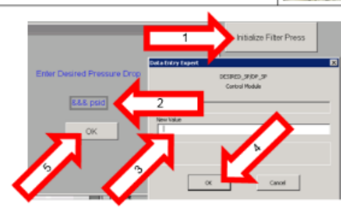
2. Conducting an Experimental Run

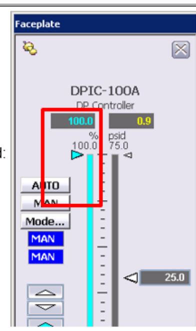
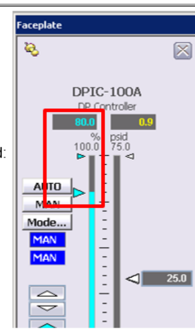
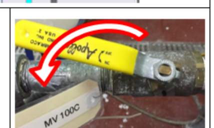
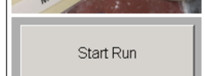
Step	Action and Rationale	Image
11	After 5 minutes of the recycling process, when the tank is well-mixed, sample the feed in the tank for weight percent solids (wt% S). See the Microwave Dryer SOP. Make sure to turn off the agitator when taking a sample and turn it back on before taking the next sample. To get the most accurate results, take at least 3 samples, each from about 12 inches below the surface level of the mixture. Use the silver scoop to take the samples.	
12	If the slurry samples are between 10 and 12 wt% S, proceed to the next step. If not, add solid calcium carbonate or water until the desired solid percentage is reached. Record the concentration in the lab notebook.	
13	Record the initial reading of the volume on the instrument display of the Magnetic Flow Meter FIT-100A.	

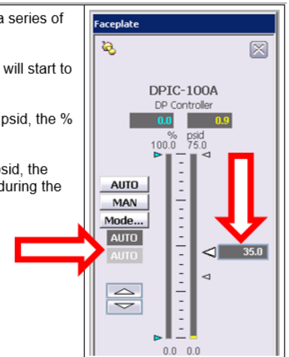
14	Wet and clean the filter canvas (canvas should be dripping slightly) and the frame. Weigh together.	
15	Clean, dry, and weigh the metal pan.	

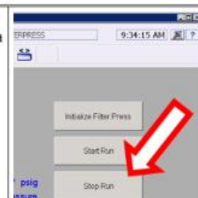
STANDARD OPERATING PROCEDURE DOCUMENT

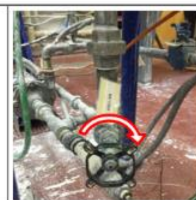
16	Secure the wet filter canvas and frame in the press. The hole in the frame with the slit must be in line with the hole in the canvas and the feed stream passing through MV-100C to allow proper flow. See below. Also make sure that there are no strings from the canvas between the canvas and any other frame. The corners of the canvas tend to flip up, so check that they are not between the frame and canvas. Pull the canvas tightly from the bottom while pushing the next plate against the frame so the canvas does not slide (this works best with a person on either side of the filter press). Correct Hole Placement:  Poor Hole Placement: 	 
17	Slide the other frames towards the canvas side of the filter press, two at a time. Tighten the filter press by turning the crank at the end clockwise. Use the pipe to tighten the filter press as much as possible. DO NOT STAND OR JUMP ON THE PIPE.	

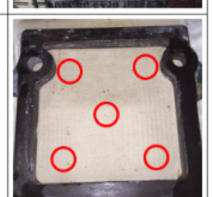
18	Place the metal pan under the frame and canvas to catch the filtrate runoff during the run.	
19	Return to the DeltaV operating station. Click the "Initialize Filter Press" button found in the upper right portion of the Filter Press operating screen (**1**). In the pop-up window, enter the desired set point for the pressure drop (2). The pressure drop may be set between 30 and 45 psid. Select "OK" and the pop-up window will close.	

20	**IMPORTANT:** After initialization, look at the faceplate. The % opened (teal bar) should change to **80%** after a few seconds. If it remains at 100%, the initialization has failed. Repeat step 19. **DO NOT MOVE ON TO STEP 21 UNLESS THE INITIALIZATION HAS SUCCEEDED.** Note that in the controller faceplate the set point value will continue to show 25.0 even though a different value for the set point was eneterd in the dialog box. This is normal because the controller is still in MAN mode. The set point will change to the entered value when the controller mode changes to AUTO. This is done automatically by the control algorithm.	Failed:  Succeeded: 
21	*This step requires the simultaneous actions of two people. Read the entire step before proceeding.* To begin running the experiment, one person will open manual valve MV-100C. Simultaneously, another person should click the "Start Run" button on the right side of the Filter Press operating screen. Select "Yes" once you are sure you want to start the run. **Important:** While manual valve MV-100C is being opened, do not stand by the side of the filter press. If anything is obstructing the seal between the canvas and the frame, feed will gush out of both sides of the frame. If this happens, • Close MV-100C immediately • Press the "Stop Run" button on the right side of the Filter Press operating screen • Call Mr. Cunning	 

22	Monitor the information displayed on the Controller Faceplate. The control algorithm executes a series of steps. The % open will start to decrease in 1% increments. At the same time, the pressure differential will start to increase. When the difference between the pressure differential and the desired set point is less than 10 psid, the % open will start to decrease in 0.5% increments. When the difference between the pressure differential and the desired set point is less than 1 psid, the mode will change from MAN to AUTO and the set point value will change to the value entered during the initialization step.	
23	Open the Process History View (PHV) file for the experiment. See GUI section for details.	
24	Monitor the flow rate of filtrate during the run. The flow rate can be monitored on both the process history view and the operator interface.	

25	Move the mouse frequently so that the monitor does not shut down automatically. When the filtrate flow rate falls below **0.01 gal/min**, press the "Stop Run" button. The control algorithm will automatically initiate a sequence of events. The controller will automatically be put in manual mode, the pressure drop will no longer be maintained, and the control valve FIC-100A will be set to 100% open. Note: If you are doing two successive runs, you must wait at least one minute after pressing "Stop Run" before initializing the second run.	
26	Close manual valve MV-100C once the control valve FIC-100A has reached 100% open.	
27	Turn off the pump using the "Pump Power" switch.	

28	Close MV-100A completely.	
29	Wait until the pressure drop falls to around 2 psid. Then, open the filter press by turning the crank counterclockwise. Some filtrate runoff may spray from the side of the press when you do this. Try not to allow any unfiltered slurry to enter the pan.	
30	Slide the empty frames away from the frame and canvas containing the cake (easier with a person on each side). Pull the frame and canvas up slowly, holding the bottom so as to catch any runoff.	

31	Allow the canvas and frame to drip into the pan until the drips have mostly stopped. Weigh the pan with the filtrate runoff and take 3 samples to find the % solids, and then pour the runoff back into the feed tank. (Turn off the agitator while the feed tank lid is open.) This is to ensure that the filtrate is composed of nearly pure water.	
32	Weigh the frame, canvas, and filter cake together.	
33	Take 5 samples from different locations on the filter cake and analyze them for % solids. Each sample should be about the size of a teaspoon. The capacity of the microwave dryer is 5g maximum, 3g preferred. See the Microwave Dryer SOP. (Tip: To make sure that your samples don't dry out before you can analyze them, place them in sealed zip-lock bags, which may be obtained in Mr. Cunning's office if there are none by the sink.)	

34	Return the filter cake and the analyzed samples to the feed tank in small chunks so as not to interfere with the agitator. (Turn off the agitator while the feed tank lid is open.)	
35	Record the final reading of the volume totalizer on FIT-100A. Find the difference between the initial reading (Step 13) and the final reading. Compare this to the DeltaV data. Note that DeltaV does not begin recording the volume until the pressure drop is within 1 psid of the set point.	

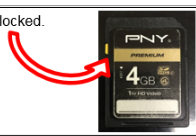
3. Area Cleanup

Step	Action and Rationale	Image
36	Wash the frame, canvas, and pan. If the floor has been dirtied by spraying or dripping, mop up the area.	
37	Pump the filtrate water back to the feed tank: • Make sure that MV-100A is closed • Open MV-100F • Turn on the pump As soon as the water has been returned, turn off the pump and close MV-100F. If MV-100F is left open when the pump is turned off, slurry may be siphoned from the feed tank into the filtrate tank.	 

38	Turn off the cooling water (MV-100H) and shut off the agitator if it is on.	

DATA RETRIEVAL AND TRANSFER FROM THE DELTAV HISTORIAN

Step	Action and Rationale
1	From the Process History View, determine the start and end times for data retrieval.
2	Open the Excel file “Data Template - Filter Press.xlsx” located on the Desktop.
3	Immediately save the file under a different unique name.
4	You may want to create a folder on the desktop in which to save the data during the course of the experiment. As a suggestion, the folder name could be “Group xx, Fall/Spring/Winter yyyy”.
5	Enter the correct start and end times in cells I1 and K1. Note that the times must be entered in cells I1 and K1, not cells D1 and F1.
6	Select cells H1 through K1; copy and paste them into cells C1 through F1. It is important to copy and paste all the cells at the same time to ensure proper operation of the data retrieval routine.
7	Watch the progress status at the bottom of the screen. Depending on the amount of data selected for retrieval, the process may take a few minutes.
8	Scroll down to verify that all the requested data have been retrieved. If this is not the case, contact your instructor.
9	Save the file.
10	Go to the “Data – Values” worksheet and make sure that it is blank. If not, delete all the contents.
11	Go back to the “Data – Links” worksheet. Select and copy all the data in it.
12	Return to the “Data – Values” worksheet and Special Paste the data as Values. Pasting the data as Values is very important. If not done, the data will not show up when you transfer the file to your laptops.
13	Save and close the file.
14	Obtain an SD card from Mr. Cunning. You should not use personal thumb drives or SD cards to transfer the data to your laptops.

Step	Action and Rationale
15	Verify that the card is unlocked. 
16	Copy the file from the desktop to the SD card reader. The SD card reader is Drive E under My Computer.
17	Lock the SD card before inserting it into your laptop. Locking the SD card is very important. It prevents the transfer of computer viruses from the students’ laptops to the DeltaV control system. The DeltaV control system does not have virus protection.
18	Copy the file from the SD card to your laptop.
19	Unlock the card and return it to Mr. Cunning.

DELTAV LOGOFF

Proper log-off from DeltaV is very important. If you do not log off properly, you will be blocking the remote session and the group using the equipment after you may not be able to log on to the DeltaV system.

Step	Action and Rationale
1	Close DeltaV Operate (Run) by clicking the “X” in the top right corner of the computer screen.
2	Close Process History View by clicking the “X” in the top right corner of the computer screen.
3	Close any other applications.
4	Click on “Start” and select “Log Off Sperry Filter Press”.
5	Allow the Remote Desktop application to close.
6	Click on “Start” and select “Lock”.
7	Do not turn the computer off!
8	Turn the monitor off.

REVISION HISTORY AND APPROVALS

1. Revision History

Rev.	Effective	Summary of Changes	Author(s)
1.0	29 Aug 2015	New Document	Megan Liebman
1.1	8 Feb 2016	Updated the location of SDS documents	Megan Liebman

2. Approvals

Title	Name	Signature	Date
Responsible Faculty			
Laboratory Technician	Frank Cunning		
Lab Course Coordinator			
Department Head	Adam Nolte		

STANDARD OPERATING PROCEDURE CHECKLIST

Area 100 – Filter Press	Revision 1.1 Effective 8 February 2016	Page **1** of **7**

PURPOSE

The purpose of this document is to:

- Provide a concise list of instructions that can be easily understood and followed by students who are already familiar with the experiment.
- Ensure that all experimental steps are completed and executed in the correct sequence.
- Provide accountability for the correct execution of the experiment.
- Document the operating conditions.
- Create a permanent record for the execution of the experiment.

Refer to the full SOP document for details and explanations on the individual steps.

GENERAL INSTRUCTIONS

- Print a hardcopy of this document and complete the signatures section prior to the start of the experiment.
- Have a copy of the full SOP document readily available.
- If you are running the experiment for the first time, follow the full SOP document.
- As soon as a step is completed, the person responsible for the execution of the step must initial to indicate completion.
- Initial each step as you go through the procedure.
- Upon completion of the experiment, file the document in the appropriate folder.
- A separate checklist is required for each experimental run.

SIGNATURES

Academic Year:____________ Group Number:__________________________

Quarter:__________ Instructor:______________________________

Student Name	Signature	Initials	Date

SAFETY PRECAUTIONS

Kinetic Hazard: Shut down the agitator prior to opening the feed tank lid.

Pinching Hazard: Use caution when tightening or loosening the plate and frame assembly.

Chemical Hazard: Calcium carbonate can dry and crack skin. Use gloves for protection.

Chemical Hazard: Calcium carbonate particles can cause eye damage if trapped under a contact lens. Use goggles if wearing contact lenses.

Refer to the Risk Analysis document for details.

DELTAV LOGON

Step	Action	Relevant Data and Units	Operator Initials
1	Turn on the Remote Operator station.		
2	Log on to the computer.		
3	Open the Remote Desktop application.		
4	Connect to the default IP address.		
5	Wait until DeltaV initializes.		
6	Launch DeltaV Operate.		
7	Acknowledge the license agreement.		
8	Open the Filter Press operating station.		
9	Ignore flashing alarms.		

EQUIPMENT PREPARATION

Step	Action	Relevant Data and Units	Operator Initials
1	Check that FIC-100A is fully open.		
2	Check that these valves are fully open: • MV-100B • MV-100D • MV-100I		

3	Check that these valves are fully closed: • MV-100A • MV-100C • MV-100E • MV-100F • MV-100G • MV-100J		
4	Check the level of water in the feed tank.		
5	Use the broom handle or plastic paddle to fully suspend all settled chalk in the feed tank.		
6	Open MV-100H.		
7	Turn on the agitator.		
8	Open MV-100A.		
9	Turn on the pump.		

CONDUCTING AN EXPERIMENTAL RUN

Step	Action	Relevant Data and Units	Operator Initials
10	After 5 minutes, sample the feed tank slurry for wt% S.		
11	If the slurry samples are between 10 and 12 wt% S, proceed to the next step. If not, add solid calcium carbonate or water until the desired solid percentage is reached.	Feed Concentration: 1) 2) 3)	
12	Record the initial volume reading of FIT-100A.	Initial Volume:	
13	Wet, clean, and weigh the filter canvas and frame.	Initial Canvas and Frame Weight:	

STANDARD OPERATING PROCEDURE CHECKLIST

Area 100 – Filter Press	Revision 1.1 Effective 8 February 2016	Page 4 of 7

14	Clean, dry, and weigh the metal pan.	Initial Pan Weight:	
15	Secure the wet filter canvas and frame in the press.		
16	Close the filter press.		
17	Place the metal pan under the frame and canvas.		
18	Initialize the filter press and enter the desired set point for the pressure drop.	Pressure Drop Set Point:	
19	Check that the initialization has succeeded.	Number of Initialization Attempts:	
20	Open MV-100C. Simultaneously, start the run in DeltaV.		
21	Monitor the information displayed on the Controller Faceplate.		
22	Open the PHV file.		
23	Monitor the flow rate of filtrate.		
24	When the filtrate flow rate falls below 0.01 gal/min, stop the run.		
25	Close MV-100C.		
26	Turn off the pump.		
27	Close MV-100A.		
28	Once the pressure drop falls to 2 psid, open the filter press.		
29	Remove the frame and canvas containing the cake.		

30	Weigh the pan with the filtrate runoff and take 3 samples to find the wt% S, then pour the runoff back into the feed tank.	Final Pan Weight: Runoff Concentration: 1) 2) 3)	
31	Weigh the frame, canvas, and filter cake together.	Final Canvas and Frame Weight:	
32	Take 5 samples from different locations on the filter cake to find the wt% S.	Cake Concentration: 1) 2) 3) 4) 5)	
33	Return the filter cake and samples to the feed tank.		
34	Record the final volume reading of FIT-100A. Find the difference between the initial reading and the final reading. Compare this to the DeltaV data.	Final Volume: Final-Initial Difference:	

AREA CLEANUP

Step	Action	Relevant Data and Units	Operator Initials
35	Wash the frame, canvas, and pan. Mop up the area.		
36	Pump the filtrate water back into the feed tank. • Make sure MV-100A is closed • Open MV-100F • Turn on the pump • Wait for filtrate collection tank to empty • Turn off the pump • Close MV-100F		
37	Close MV-100H and shut off the agitator.		

DATA RETRIEVAL AND TRANSFER FROM THE DELTAV HISTORIAN

Step	Action	Relevant Data and Units	Operator Initials
1	Determine the start and end times for data retrieval.	Start Time: End Time:	
2	Open "Data Template – Filter Press.xlsx".		
3	Immediately save the file under a different unique name.	File Name:	
4	If not previously done, create a folder on the desktop.		
5	Enter the correct start and end times in cells I1 and K1.		
6	Select cells H1 through K1; copy and paste them into cells C1 through F1.		
7	Wait until the status at the bottom of the screen indicates that the transfer is complete.		
8	Verify that all requested data have been retrieved.		
9	Save the file.		

10	Clear all contents from the “Data – Values” worksheet.		
11	Select and copy all data from the “Data – Links” worksheet.		
12	Special Paste the data as Values in the “Data – Values” worksheet.		
13	Save and close the file.		
14	Obtain an SD card from Mr. Cunning.		
15	Verify that the card is unlocked.		
16	Copy the file from the desktop to the SD card reader.		
17	Lock the SD card before inserting it into your laptop.		
18	Copy the file from the SD card to your laptop.		
19	Unlock the card and return it to Mr. Cunning.		

DELTAV LOGOFF

Step	Action	Relevant Data and Units	Operator Initials
1	Close DeltaV Operate (Run).		
2	Close Process History View.		
3	Close any other applications.		
4	Log off from DeltaV.		
5	Allow the Remote Desktop application to close.		
6	Lock the computer.		
7	Do not turn the computer off!		
8	Turn the monitor off.		

APPENDIX D: Corning Column Standard Operating Procedure and Checklist

STANDARD OPERATING PROCEDURE DOCUMENT

Area 200 – Corning Column	Revision 1.1 Effective 8 February 2016	Page 1 of 36

Supersedes:	Revision 1.0
Purpose:	The purpose of this document is to describe the setup and operation of the Corning Column experiment. The Corning Column experiment is used in the undergraduate Chemical Engineering Laboratory courses to study the unit operation of binary distillation. **In-Scope** This document applies to the use of the Corning Column experiment in the context of the undergraduate Chemical Engineering Laboratory courses. **Out-of-Scope** This document does not apply to uses of the Corning Column experiment outside of the undergraduate Chemical Engineering Laboratory courses.
Area(s) Involved:	Chemical Engineering Laboratory, Area 200 – Corning Column
Reasons for Revision:	Updated the location of SDS documents.

STANDARD OPERATING PROCEDURE DOCUMENT

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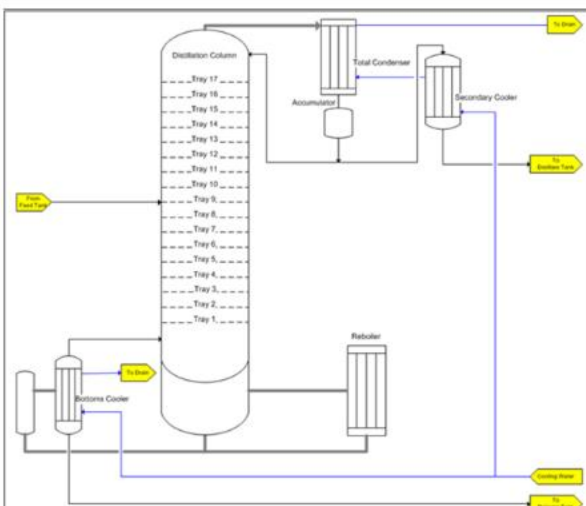
TABLE OF CONTENTS

EQUIPMENT OVERVIEW

1. Experimental Setup

The Corning column setup consists of a Corning glass distillation column, a feed tank, a bottoms tank, a distillate tank, and two control panels. The distillation column is made from Corning glass to allow viewing of the column internals. A mixture of isopropanol and isobutanol is separated in the column. The column contains seventeen Teflon sieve trays of one-inch thickness and six-inch diameter. The trays are spaced ten inches apart. The thermosiphon reboiler has a six-inch interior diameter, a three-inch diameter crossover section, and a 1.5-meter connection to the column. All cooling is done via cooling water. The total condenser at the top of the column is open to atmosphere. The condensed vapor is collected in the accumulator. Based on the specified reflux ratio, part of the condensed distillate is returned back to the column as reflux and the rest is collected as distillate product. Two coolers, one for the distillate and one for the bottoms, reduce the temperature of the product streams before they are sent back into the corresponding storage tanks. A simplified schematic of the column can be seen on the following page. Refer to the P&ID document for details. The DeltaV process control and data acquisition system enables automated operation of the system and records all measured process variables.

If prompted for "**Upload**" by the control system, ALWAYS choose **Cancel**.



Simplified Schematic. ***Refer to the P&ID document for details.***

2. Operational Ranges

Feed Composition: 0.4 to 0.6 mol % isopropanol
Feed Tray: 7, 9, or 11
Reflux Ratio: 3.0 to 3.8

3. Experimental Progression

To start up the column, manual adjustments are made to the valves and switches to turn on electrical power and cooling water. The control system (DeltaV) executes a startup sequence to bring the column to normal operation. After that it maintains the set reflux ratio and monitors the process variables. A steady state indicator is activated when the rate of change for selected parameters falls below a threshold level. After steady state is indicated, samples are collected from the feed, distillate, and bottoms streams. The samples are analyzed while the column is being shut down. Typical times are 0.5 hours for startup, 2.5 hours to reach steady state and 0.5 hours for shutdown.

SAFETY PRECAUTIONS

Height Hazard: When accessing the upper level of the column, there is a risk of falling or dropping something onto a person below.

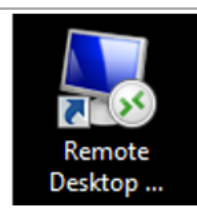
Chemical Hazard: Isobutanol is hazardous in cases of skin or eye contact (irritant), inhalation, or ingestion. Isobutanol is highly flammable.

Chemical Hazard: Isopropanol is hazardous in cases of eye contact (irritant), inhalation, or ingestion. Isopropanol is highly flammable.

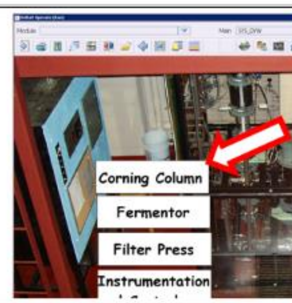
Review the SDS document for each chemical prior to conducting any experimental runs. SDS documents are located in an online database; the link is provided in the CHE Laboratory page on Moodle.

Refer to the Risk Analysis document for details.

DELTAV LOGON

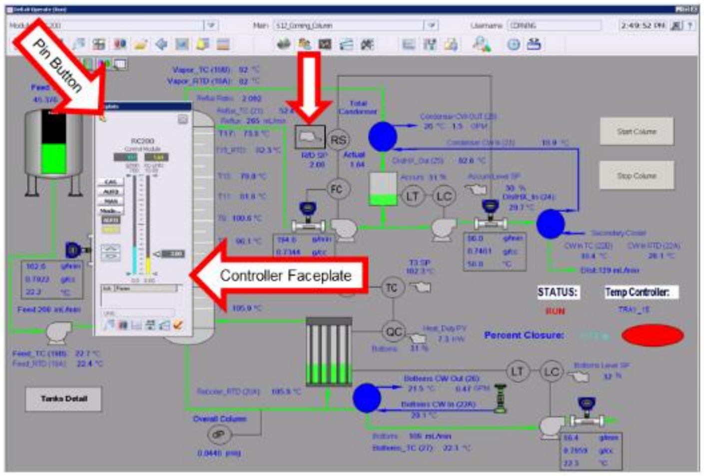
Step	Action and Rationale	Image
1	Turn on the Remote Operator station CHELAB-7 next to the experimental setup. Note: Normally the computer will be on, so only the monitor will need to be turned on.	
2	Log on to the computer: • Username: .\che (default) • Password: student	
3	Double-click the "Remote Desktop …" icon on the desktop.	

4	Connect to the default IP address (192.168.1.201): • Username: corning (default) • Password: corningcolumn	
5	Wait for approximately 30 seconds, until DeltaV initializes.	
6	When the FlexLock ribbon appears, click on the "DeltaV Operate" button.	
7	When the DeltaV Simulate window appears, select "OK" to acknowledge the license agreement.	

8	Click on the "Corning Column" button on the DeltaV main screen.	
9	Ignore any alarms that may be flashing at the bottom of the screen.	

GRAPHICAL USER INTERFACE (GUI)

1. Experiment Main Display



To display the controller faceplate, click on the image of the hand labeled "R/D SP" and drag the faceplate to the position shown above. Pin the faceplate to the display using the Pin button.

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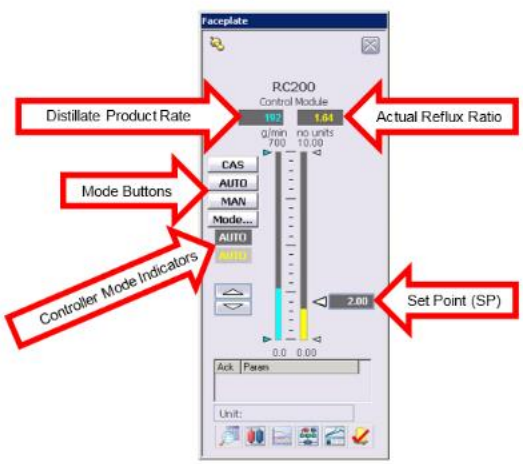
2. Controller Faceplate

The Controller Faceplate shows and allows access to the most important operating parameters of the control algorithm.

The callouts in the printscreen below describe some of the most important fields in the Reflux Ratio Controller Faceplate.



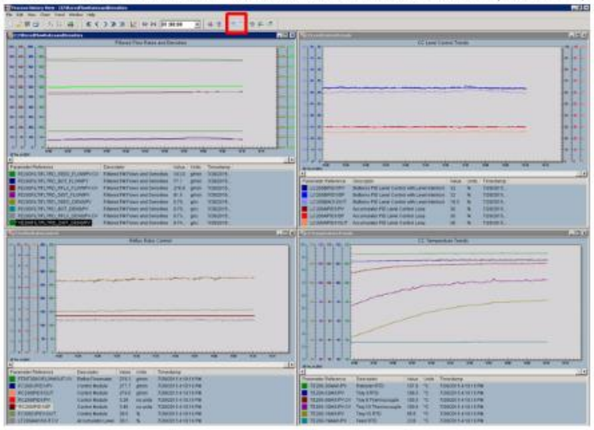
3. Process History View (PHV) Application

The Process History View (PHV) is an application that provides real-time and historical data in a chart recorder graphical format. It is used to monitor the progress of the experiment.

To access the Process History View utility, switch to Desktop view and double-click on the Process History View icon (1). One of the charts will load automatically, but you will need to open the three others. Go to "File" and select the other three from the pane with recently-accessed files (2):

- CCRefluxRatioControl
- CCFilteredFlowRatesandDensities
- CCLevelControlTrends
- CCTemperatureControlTrends

Click one of the "Tile Windows" buttons in the ribbon near the center of the screen (boxed in red, below) to view all charts simultaneously.



(1)

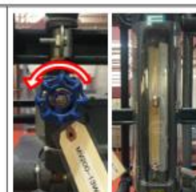
(2)

PROCEDURAL DESCRIPTIONS

1. Startup

Step	Action and Rationale	Image
1	Review the appropriate SDS documents and the chemical agent release response plan for this experiment.	
2	On the blue panel next to the distillation column, ensure that the circuit breakers for the auxiliary pump and the feed preheater are in the off position.	
3	Turn on only the following circuit breakers: • Main • Control Pumps • Auto Controls Power • Temp • Heater Element	

4	Fully open the main water valve MV200-11W located on the back side of the column.	
5	Open the bottoms cooling water valve MV200-12W to a flow rate of approximately 0.5 gpm, as read on the right-hand flowmeter FIT200B.	
6	Open the condenser cooling water valve MV200-13W to a flow rate of approximately 0.8 gpm, as read on the left-hand flowmeter FIT200A.	

7	After waiting 10-15 seconds for any air that may be trapped in the line to escape, increase the flow rate to 1.5 gpm. Confirm this flow rate with the rotameter. Monitor this flow rate throughout the day.	
8	Verify that all toggle switches on the blue panel are in the off position (middle). These switches allow you to select an operation mode of manual (left), off (middle), or automatic (right).	
9	Verify that the rotary knob for the heater, located above the toggle switches, is off (full counterclockwise position). Note: Leave the rotary knob for the heater in the full counterclockwise position for the duration of the laboratory period.	

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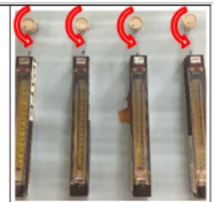
10	Verify that the rotary knobs for the pumps, located above the pump rotameters, are off (full counterclockwise position).	
11	Verify that MV200-06, near the bottom of the feed tank, is open.	

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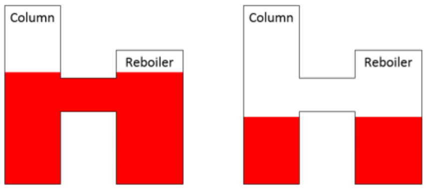
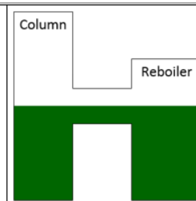
12	Go to the upper level of the column. Open MV200-08 and close MV200-10.	
13	Open the valve corresponding to the feed tray to be used and make sure that the other two valves are closed. Your choices are tray 7, 9, or 11. Tray 9 is the most commonly used feed location.	

14	Check to make sure that the liquid level in the reboiler is within the crossover region, as shown to the right. If this is not the case, i.e. the level is too high or too low, as shown below, contact your instructor or Mr. Cunning. Do not proceed further. 	
15	Switch all of the toggle switches on the blue panel to automatic position (right).	

16	Turn on the vent EF-1. The switch is located on the East wall behind the column.	

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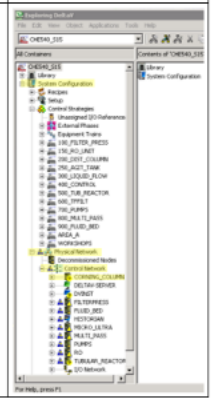
2. Executing a Run

Step	Action and Rationale	Image
17	In the DeltaV Operate window, select the "DeltaV Utilities" icon in the central grouping of icons in the ribbon across the top of the screen. Then, select "DeltaV Explorer", the icon second from the left. **Note: If prompted for upload, choose Cancel.**	
18	Locate the Corning Column by following this file path: System Configuration > Physical Network > Control Network. Right-click on "CORNING_COLUMN" and select Download > Controller. If prompted for Upload, ALWAYS choose Cancel.	

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19	Confirm Total Download Note: If prompted for upload, choose Cancel.	
20	Call your instructor if the Download is completed with errors or warnings After successful download, click Close and exit the DeltaV Explorer.	
21	On the right-hand side of the DeltaV Operate screen, check that the Temp Controller is set to TRAY_15.	
22	Click on the Start Column button on the right-hand side of the DeltaV Operate screen. The status should change from IDLE to STARTUP.	

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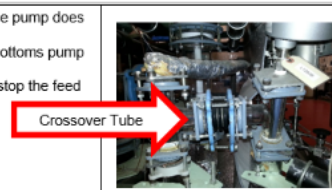
23	Monitor the crossover tube level to the bottoms pump to make sure the pump does not run dry or the column becomes flooded. • If the level falls below the crossover point, manually stop the bottoms pump using the toggle switch. • If extensive flooding is observed in the second tray, manually stop the feed pump using the toggle switch. When the crossover level returns to normal, put the toggle switch back to AUTO position.	 
24	To monitor the various outputs of the column, access the Process History View using the procedure described in GUI section of the SOP.	
25	Once the column has run long enough to heat up, the status of the column will change from STARTUP to RUN. The details of the startup sequence are described in Appendix 2.	

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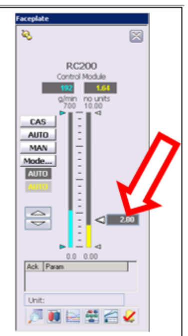
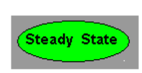
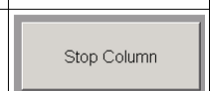
26	Set the reflux ratio to the value specified by your instructor. The set point can be changed only after the column status has changed to RUN. Access the Reflux Ratio controller faceplate using the procedure described in GUI section of the SOP. Enter the desired value for the reflux ratio in the box with the white text on the right-hand side of the controller faceplate.	
27	After the column reaches the set point reflux ratio and stabilizes to steady state, the status of the column will change from red RUN to green STEADY STATE and the oval steady-state indicator will change from red to green. Note that the steady state indicator takes into account only the rate of change of levels, flowrates, and temperatures. The ultimate test for steady state is the component material balance which must be performed for every run. The details of the steady state algorithm are described in Appendix 1.	

3. Sampling

Step	Action and Rationale	Image
28	When it is determined that steady-state operating conditions have been met, samples of the feed, distillate, and bottoms product can be taken. The sampling ports are located below the rotameter panel.	
29	Obtain clean sampling bottles. Use 15 mL bottles.	
30	Prepare an ice bath to put the bottles in immediately after sample collection.	
31	Slowly open the valve to the sampling port and drain a small amount of product into a beaker. Drain just enough to remove what may have been in the sampling port piping.	
32	Collect the sample. Fill the bottle to the top to avoid flashing in the headspace of the bottle. Close the sampling port, and immediately screw the cap on the bottle. When shutting the valve, do not overtighten or the valve can become damaged. It is okay if a small amount of product drips onto the metal tray below the sampling ports.	
33	Put the bottle in the ice bath.	
34	Analyze the samples with the Anton Paar Density Meter to determine their composition. See the SOP for the Anton Paar Density Meter.	

4. Shutdown

Step	Action and Rationale	Image
35	After all of the samples have been taken for the day, click on the Stop Column button on the right-hand side of the DeltaV Operate screen. The status should change to SHUTDOWN. Feed and reflux streams should immediately halt. The reading for the reflux stream will not immediately show a reading of zero due to the signal processing filter. The bottoms and distillate streams will automatically shut down once they have reached a preset level.	
36	Wait for the system status to change to IDLE.	
37	Log off from DeltaV. Follow the DeltaV Logoff procedure.	
38	Set all toggle switches to the off position.	

39	On the upper level of the column, close valve 8 and open valve 10.	
40	Check to make sure that the feed stage is set to Stage 9 by opening valve 9 and closing valves 7 and 11.	
41	Increase the flow rate of cooling water through the bottoms cooler to approximately 1.5 gpm, as read on the right-hand flowmeter FIT200B, by adjusting the bottoms cooling water valve MV200-12W.	 

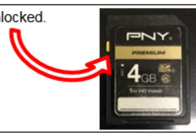
42	Turn the bottoms pump toggle switch to manual and turn the bottoms pump rotary knob to 50% flow, as indicated by the rotameter.	 
43	Verify that the thermocouple dial is set to position 19 (bottoms temperature).	

44	When thermocouple 19 (bottoms temperature) drops to 185ºF, turn the pump to full power.	
45	Watch the bottoms crossover level to avoid running the bottoms pump dry. If the level runs low call your instructor or the laboratory technician.	
46	Once the temperature of the bottoms drops to 150ºF, turn off the bottoms pump toggle switch.	

47	Slowly, shut the cooling water valves: • MV200-11W • MV200-12W • MV200-13W	
48	Turn off the heater and main power circuit breakers.	
49	Turn off the vent EF-1. The switch is located on the East wall behind the column.	

DATA RETRIEVAL AND TRANSFER FROM THE DELTAV HISTORIAN

Step	Action and Rationale
1	From the Process History View, determine the start and end times for data retrieval.
2	Open the Excel file “Data Template - Corning Column.xlsx” located on the Desktop.
3	Immediately save the file under a different unique name.
4	You may want to create a folder on the desktop in which to save the data during the course of the experiment. As a suggestion, the folder name could be “Group xx, Fall/Spring/Winter yyyy”.
5	Enter the correct start and end times in cells H1 and J1. Note that the times must be entered in cells H1 and J1, not cells C1 and E1.
6	Select cells G1 through J1; copy and paste them into cells B1 through E1. It is important to copy and paste all the cells at the same time to ensure proper operation of the data retrieval routine.

7	Watch the progress status at the bottom of the screen. Depending on the amount of data selected for retrieval, the process may take a few minutes.
8	Scroll down to verify that all the requested data have been retrieved. If this is not the case, contact your instructor.
9	Save the file.
10	Go to the "Data – Values" worksheet and make sure that it is blank. If not, delete all the contents.
11	Go back to the "Data – Links" worksheet. Select and copy all the data in it.
12	Return to the "Data – Values" worksheet and Special Paste the data as Values. Pasting the data as Values is very important. If not done, the data will not show up when you transfer the file to your laptops.
13	Save and close the file.
14	Obtain an SD card from Mr. Cunning. You should not use personal thumb drives or SD cards to transfer the data to your laptops.
15	Verify that the card is unlocked. 
16	Copy the file from the desktop to the SD card reader. The SD card reader is Drive G under My Computer.
17	Lock the SD card before inserting it into your laptop. Locking the SD card is very important. It prevents the transfer of computer viruses from the students' laptops to the DeltaV control system. The DeltaV control system does not have virus protection.
18	Copy the file from the SD card to your laptop.
19	Unlock the card and return it to Mr. Cunning.

DELTAV LOGOFF

Proper log-off from DeltaV is very important. If you do not log off properly, you will be blocking the remote session and the group using the equipment after you may not be able to log on to the DeltaV system.

Step	Action and Rationale
1	Close DeltaV Operate (Run) by clicking the "X" in the top right corner of the computer screen.
2	Close Process History View by clicking the "X" in the top right corner of the computer screen.
3	Close any other applications.
4	Click on "Start" and select "Log Off corning".
5	Allow the Remote Desktop application to close.
6	Click on "Start" and select "Lock".
7	Do not turn the computer off!
8	Turn the monitor off.

REVISION HISTORY AND APPROVALS

1. Revision History

Rev.	Effective	Summary of Changes	Author(s)
1.0	29 Aug 2015	New Document	Megan Liebman
1.1	8 Feb 2016	Updated the location of SDS documents	Megan Liebman

2. Approvals

Title	Name	Signature	Date
Responsible Faculty			
Laboratory Technician	Frank Cunning		
Lab Course Coordinator			
Department Head	Adam Nolte		

APPENDIX

1. Steady State Algorithm

In an effort to numerically determine when the Corning column is at steady state using DeltaV, a derivative-based approach was used and resulted in the following programmed module (CORNING-COLUMN/CC_STEADYSTATE/CC_DERIVATIVES), Figure A1-1.

In the module, there are eight input parameters; accumulator level, bottoms level, reboiler temp, tray 3 temp, tray 15 temp, tray 8 temp, distillate flow rate, and reflux flow rate. Each input is run through a series of four blocks which effectively take the derivative of the input stream, Figure A1-2. Since there is no derivative block, the change in input divided by the change in time is used as a derivative approximation. The input parameter is specified in the ACCUM_ block which serves as the input to the FLTR block. The FLTR block filters the data to get rid of noise and has a set filter time of 60 sec. The FLTR block has two outputs; one output goes directly to the SUB block while the other output goes to the DT block. In the DT or dead time block, the filtered value is held for the set time of 120 sec before serving as the second input into the SUB block. In the SUB block, the direct output of the FLTR block is subtracted from the output of the DT block.

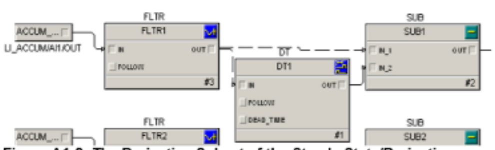


Figure A1-2: The Derivative Subset of the Steady State/Derivative Module Schematic

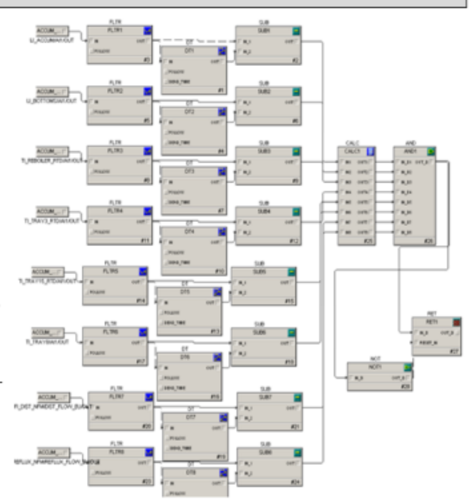


Figure A1-1: DeltaV Control Steady State/Derivative Module Schematic

The outputs from the eight SUB blocks are sent to the CALC block to compare derivative values with programmed range values, Figure A1-3. The following code was programmed into the CALC block:

IF 'input' ≤ **Value** THEN 'output' := TRUE;
ELSE 'output' := FALSE;
ENDIF;

The **Value** used in this code varied for each parameter according to Table A1-1. TRUE and FALSE are represented by the numbers 1 and 0, respectively. The eight outputs from the CALC block are then sent to the AND block which adds them together. If all statements are TRUE, then the output is also TRUE or 1. If a single input is FALSE, then the output is also FALSE or 0. This value is then used as the input for both the RET and the NOT block. The NOT block simply reverses the input: if the input is TRUE, the output is FALSE and vice versa. The final block, RET, looks at the output of the AND block. When the input to the block is TRUE, a five minute timer is started so that if the input remains TRUE for five minutes then the output will also display TRUE indicating the column is at steady state. If the input changes to FALSE, the timer restarts. When the output from the NOT block turns FALSE after the RET output is TRUE, the RET block is reset and the timer started once again.

Table A1-1: Experimentally Determined Max Absolute Values for the Derivative Fluctuation of Steady State Parameters

Parameter	Max Absolute Value
Accumulator Level	0.1167*T - 9.05
Bottoms Level	1
Reboiler Temp	0.1
Tray 3 Temp	0.2
Tray 15 Temp	0.0317*T – 2.555
Tray 8 Temp	0.4
Distillate Flow Rate	5
Reflux Flow Rate	3

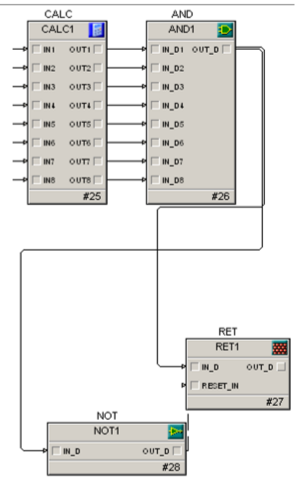


Figure A1-3: The Steady-State Subset of the Steady State/Derivative Module Schematic

2. Startup Protocol

In order to safely start up the Corning distillation column and speedily bring it to its first steady state using tray 15 temperature control application of DeltaV, the program, CORNING-COLUMN/CC2_TRAY15/SFC_CC_START, was previously programmed, Figure A2-1. A printing of the exact code for the entire module can be made from DeltaV Control Studio. A description of the function of each block follows.

INITIALIZE
- PID controllers for the accumulator level, bottoms level, heat duty, and tray 15 temperature are set to manual mode.

SET_SP_OUTPUT1
- Tray 15 temperature and head duty controller outputs are set to zero.
- The initial controller settings for the heat duty and tray 15 temperature are specified (Table A2-1).

SET_SP_OUTPUT4
- The initial controller settings for the accumulator and bottoms levels are specified (Table A2-1).

STOP_AND_WAIT
- The column status is changed to idle.
- The "START" button on the user interface screen must be selected to proceed to the next block.

RE_INITIALIZE
- The accumulator and bottoms level controllers are set to auto mode.
- The heat duty and tray 15 temperature controllers remain in manual mode.

SET_SP_OUTPUTS
- If the reboiler temperature is greater than or equal to 80, then the bottoms level set point is set to 25, else 30.
- The following control parameters are set for the bottoms level controller:
    - Gain = 12

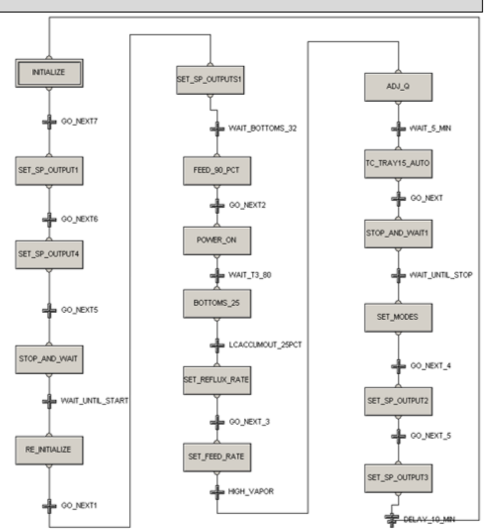


Figure A2-1: DeltaV Corning Column Startup Protocol Schematic

- Reset = 20
- PV_FTime = 10

- The accumulator level controller set point is set to 30.
- The output of the heat duty and tray 15 temperature controllers are still set to zero.
- The bottoms level must be between 22 and 32% to proceed to the next block.

FEED_90_PCT
- The tray 15 temperature controller set point is changed to 90.

POWER_ON
- If the reboiler temperature is greater than or equal to 80 then the heat duty controller set point is changed to 5.6. If not, it is changed to 8.6
- The heat duty controller is set to auto mode.
- Tray 3 temperature must be greater than or equal to 80 to proceed to the next block.

BOTTOMS_25
- The following controller settings for the bottoms are changed:
  - Gain = 8
  - Set point = 25
  - Reset = 200
- The accumulator level controller output must be greater than or equal to 25% to proceed to the next block

SET_REFLUX_RATE
- The tray 15 temperature output is set to 40.

SET_FEED_RATE
- The tray 15 feed-out set point is changed to 56.
- There is a five minute waiting period in addition to the accumulator level controller output having to be greater than or equal to 14 to proceed to the next block.

ADJ_Q
- The heat duty controller set point is changed to 6.6.
- The tray 15 controller output is changed to 20.
- There is a five minute waiting period before proceeding to the next block.

TC_TRAY15_AUTO
- Tray 15 controller is set to auto mode.
- The following bottoms level controller settings are changed:
  - Set point = 22

Table A2-1: Initial Controller Settings in Both the PID Controllers and the Startup Protocol

Parameter	Accumulator Level	Bottoms Level	Tray 15 Temperature	Heat Duty
SP	50	30	88	0
Gain	1.5	20	8	0.5
Reset	20	20	60	18
Rate	0	0	10	0
Hi	95	95	95	10
Hi-Hi	100	100	100	11
Lo	5	5	5	5
Lo-Lo	0	0	0	0
PV_FTime	6	1	10	8
SP lim Hi	80	42	95	10
SP lim Lo	10	15	75	0

- Gain = 8
- Reset = 200

STOP_AND_WAIT1
- The column status is changed to run.
- The “stop” button on the user interface screen must first be pushed before proceeding to the next block.

SET_MODES
- The heat duty and tray 15 temperature controllers are set to manual.

SET_SP_OUTPUT2
- The heat duty controller set point and output are set to zero.
- The tray 15 temperature controller set point and output are set to 88 and 0, respectively.
- The tray 15 feed-out set point is changed to zero.
- There is a 10 second waiting period before proceeding to the next block.

SET_SP_OUTPUT3
- The accumulator level controller set point is changed to 30
- The following bottoms level controller settings are changed:
  - Set point = 30
  - Gain = 20
  - PV_FTime = 1
  - Reset = 1000
- There is a ten-minute delay before the column can be started up again.

STANDARD OPERATING PROCEDURE CHECKLIST		
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PURPOSE

The purpose of this document is to:

- Provide a concise list of instructions that can be easily understood and followed by students who are already familiar with the experiment.
- Ensure that all experimental steps are completed and executed in the correct sequence.
- Provide accountability for the correct execution of the experiment.
- Document the operating conditions.
- Create a permanent record for the execution of the experiment.

Refer to the full SOP document for details and explanations on the individual steps.

GENERAL INSTRUCTIONS

- Print a hardcopy of this document and complete the signatures section prior to the start of the experiment.
- Have a copy of the full SOP document readily available.
- If you are running the experiment for the first time, follow the full SOP document.
- As soon as a step is completed, the person responsible for the execution of the step must initial to indicate completion.
- Initial each step as you go through the procedure.
- Upon completion of the experiment, file the document in the appropriate folder.
- A separate checklist is required for each experimental run.

SIGNATURES

Academic Year:____________ Group Number:_________________________

Quarter:__________ Instructor:______________________________

Student Name	Signature	Initials	Date

SAFETY PRECAUTIONS

Height Hazard: When accessing the upper level of the column, there is a risk of falling or dropping something onto a person below.

Chemical Hazard: Isobutanol is hazardous in cases of skin or eye contact (irritant), inhalation, or ingestion. Isobutanol is highly flammable.

Chemical Hazard: Isopropanol is hazardous in cases of eye contact (irritant), inhalation, or ingestion. Isopropanol is highly flammable.

Refer to the Risk Analysis document for details.

DELTAV LOGON

Step	Action	Relevant Data and Units	Operator Initials
1	Turn on the Remote Operator station.		
2	Log on to the computer.		
3	Open the Remote Desktop application.		
4	Connect to the default IP address.		
5	Wait until DeltaV initializes.		
6	Launch DeltaV Operate.		
7	Acknowledge the license agreement.		
8	Open the Corning Column operating station.		
9	Ignore flashing alarms.		

STANDARD OPERATING PROCEDURE CHECKLIST

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STARTUP

Step	Action	Relevant Data and Units	Operator Initials
1	Review the appropriate SDS documents and the chemical agent release response plan for this experiment.		
2	Ensure that the circuit breakers for the auxiliary pump and the feed preheater are in the off position.		
3	Turn on only the following circuit breakers: • Main • Control Pumps • Auto Controls Power • Temp • Heater Element		
4	Fully open MV200-11W.		
5	Open MV200-12W to a flow rate of approximately 0.5 gpm, as read on FIT200B.		
6	Open MV200-13W to a flow rate of approximately 0.8 gpm, as read on FIT200A.		
7	After waiting 10-15 seconds, increase the flow rate to 1.5 gpm. Confirm this flow rate with the rotameter. Monitor this flow rate throughout the day.		
8	Verify that all toggle switches on the blue panel are in the off position.		
9	Verify that the rotary knob for the heater is off. Note: Leave the rotary knob for the heater in the full counterclockwise position for the duration of the laboratory period.		
10	Verify that the rotary knobs for the pumps are off.		
11	Verify that MV200-06 is open.		
12	Go to the upper level of the column. Open MV200-08 and close MV200-10.		
13	Open the valve corresponding to the feed tray to be used and make sure that the other two valves are closed.	Feed Tray:	

14	Check to make sure that the liquid level in the reboiler is within the crossover region. If this is not the case, i.e. the level is too high or too low, contact your instructor or Mr. Cunning. Do not proceed further.		
15	Switch all of the toggle switches on the blue panel to automatic position.		
16	Turn on the vent EF-1.		

EXECUTING A RUN

Step	Action	Relevant Data and Units	Operator Initials
17	In the DeltaV Operate window, select the "DeltaV Utilities" icon and then the "DeltaV Explorer" icon.		
18	Locate the Corning Column by following this file path: System Configuration > Physical Network > Control Network. Right-click on "CORNING_COLUMN" and select Download > Controller. **If prompted for *Upload*, ALWAYS choose *Cancel*.**		
19	Confirm Total Download.		
20	After successful download, click Close and exit the DeltaV Explorer.		
21	On the DeltaV Operate screen, check that the Temp Controller is set to TRAY_15.		
22	Click on the Start Column button.		
23	Monitor the crossover tube level to the bottoms pump to make sure the pump does not run dry or the column becomes flooded.		
24	Access the Process History View.		
25	Once the column has run long enough to heat up, the status of the column will change from STARTUP to RUN.		
26	Set the reflux ratio to the value specified by your instructor.	Reflux Ratio:	
27	After the column reaches the set point reflux ratio and stabilizes to steady state, the status of the column will change from red RUN to green STEADY STATE and the oval steady-state indicator will change from red to green.		

SAMPLING

Step	Action	Relevant Data and Units	Operator Initials
28	When it is determined that steady-state operating conditions have been met, samples of the feed, distillate, and bottoms product can be taken.		
29	Obtain clean 15 mL sampling bottles.		
30	Prepare an ice bath to put the bottles in immediately after sample collection.		
31	Slowly open the valve to the sampling port and drain a small amount of product into a beaker. Drain just enough to remove what may have been in the sampling port piping.		
32	Collect the sample. Fill the bottle to the top to avoid flashing in the headspace of the bottle. Close the sampling port, and immediately screw the cap on the bottle. When shutting the valve, do not overtighten or the valve can become damaged. It is okay if a small amount of product drips onto the metal tray below the sampling ports.		
33	Put the bottle in the ice bath.		
34	Analyze the samples with the Anton Paar Density Meter to determine their composition.		

SHUTDOWN

Step	Action	Relevant Data and Units	Operator Initials
35	After all of the samples have been taken for the day, click on the Stop Column button on the right-hand side of the DeltaV Operate screen. The status should change to SHUTDOWN.		
36	Wait for the system status to change to IDLE.		
37	Log off from DeltaV. Follow the DeltaV Logoff procedure.		
38	Set all toggle switches to the off position.		
39	On the upper level of the column, close valve 8 and open valve 10.		
40	Check to make sure that the feed stage is set to Stage 9 by opening valve 9 and closing valves 7 and 11.		

41	Increase the flow rate of cooling water through the bottoms cooler to approximately 1.5 gpm, as read on FIT200B, by adjusting MV200-12W.		
42	Turn the bottoms pump toggle switch to manual and turn the bottoms pump rotary knob to 50% flow, as indicated by the rotameter.		
43	Verify that the thermocouple dial is set to position 19		
44	When thermocouple 19 drops to 185ºF, turn the pump to full power.		
45	Watch the bottoms crossover level to avoid running the bottoms pump dry.		
46	Once thermocouple 19 drops to 150ºF, turn off the bottoms pump toggle switch.		
47	Slowly, shut the cooling water valves: • MV200-11W • MV200-12W • MV200-13W		
48	Turn off the heater and main power circuit breakers.		
49	Turn off the vent EF-1.		

DATA RETRIEVAL AND TRANSFER FROM THE DELTAV HISTORIAN

Step	Action	Relevant Data and Units	Operator Initials
1	Determine the start and end times for data retrieval.	Start Time: End Time:	
2	Open "Data Template – Corning Column.xlsx".		
3	Immediately save the file under a different unique name.	File Name:	
4	If not previously done, create a folder on the desktop.		
5	Enter the correct start and end times in cells H1 and J1.		

6	Select cells G1 through J1; copy and paste them into cells B1 through E1.		
7	Wait until the status at the bottom of the screen indicates that the transfer is complete.		
8	Verify that all requested data have been retrieved.		
9	Save the file.		
10	Clear all contents from the "Data – Values" worksheet.		
11	Select and copy all data from the "Data – Links" worksheet.		
12	Special Paste the data as Values in the "Data – Values" worksheet.		
13	Save and close the file.		
14	Obtain an SD card from Mr. Cunning.		
15	Verify that the card is unlocked.		
16	Copy the file from the desktop to the SD card reader.		
17	Lock the SD card before inserting it into your laptop.		
18	Copy the file from the SD card to your laptop.		
19	Unlock the card and return it to Mr. Cunning.		

DELTAV LOGOFF

Step	Action	Relevant Data and Units	Operator Initials
1	Close DeltaV Operate (Run).		
2	Close Process History View.		
3	Close any other applications.		
4	Log off from DeltaV.		
5	Allow the Remote Desktop application to close.		
6	Lock the computer.		
7	Do not turn the computer off!		
8	Turn the monitor off.		

APPENDIX E: Tubular Reactor Standard Operating Procedure and Checklist

STANDARD OPERATING PROCEDURE DOCUMENT

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Supersedes:	Revision 1.0
Purpose:	The purpose of this document is to describe the setup and operation of the Tubular Reactor experiment. The Tubular Reactor experiment is used in the undergraduate Chemical Engineering Laboratory courses to study the effect of fluid flow regime on conversion. **In-Scope** This document applies to the use of the Tubular Reactor experiment in the context of the undergraduate Chemical Engineering Laboratory courses. **Out-of-Scope** This document does not apply to uses of the Tubular Reactor experiment outside of the undergraduate Chemical Engineering Laboratory courses.
Area(s) Involved:	Chemical Engineering Laboratory, Area 500 – Tubular Reactor
Reasons for Revision:	Updated the location of SDS documents.

STANDARD OPERATING PROCEDURE DOCUMENT

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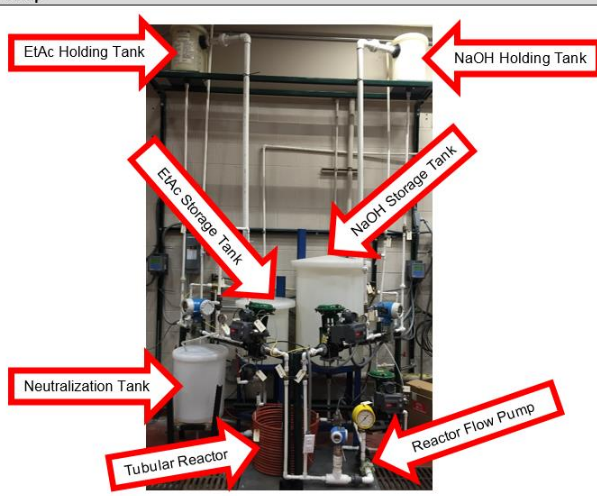
EQUIPMENT OVERVIEW

1. Experimental Setup



The Tubular Reactor setup consists of two holding tanks, two storage tanks, three pumps, a tubular reactor, and a neutralization tank.

Refer to the P&ID document for details.

2. Operational Ranges

Flow Rates through Reactor: 0.25 gpm to 1.00 gpm EtAc Concentration: 0.1 M EtAc Volume per Experimental Run: 6 gal NaOH Concentration: 0.08 M NaOH Volume per Experimental Run: 8.5 gal	Max Flow Rate during Storage Tank Fill Up: 8 gpm Holding Tanks Capacity: 4.2 gal Neutralization Tank Capacity: 30 gal

3. Experimental Progression

This experiment has a complex setup. Prior to any experimental runs, students must obtain a copy of the process and instrumentation diagram (P&ID) and trace all the lines in order to develop a good understanding of the flow of material.

Due to budgetary constraints, only one trial with chemicals is allowed. If executed properly, a single trial will produce adequate data to study the reaction in laminar, turbulent, and transitional flow regimes.

Students are required to practice the experimental procedure by running at least 3 trials with city water (no chemicals), before conducting an experimental trial with chemical reagents.

It takes 24 hours to neutralize the waste reagents collected in the waste neutralization tank. Students must return to the lab on the day following the experimental trial with chemical reagents to dispose of the waste and complete the area cleanup. This must be arranged with the instructor and the laboratory technician.

Students must contact the Chemistry Department to arrange an analytical measurement of the prepared NaOH solution.
Contact Lou Johnson, lab manager of the Chemistry Department, in FL101 or at johnson5@rose-hulman.edu.

SAFETY PRECAUTIONS

Chemical Hazard: Ethyl acetate (EtAc) is a flammable liquid and vapor. It will cause eye and skin irritation upon contact.

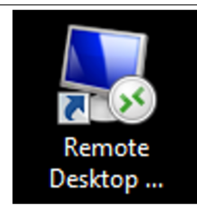
Chemical Hazard: Sodium hydroxide (NaOH) is a corrosive solid and liquid. It will cause eye and skin burns upon contact.

Environmental Hazard: Review the chemical agent release response plan document in the Appendix.

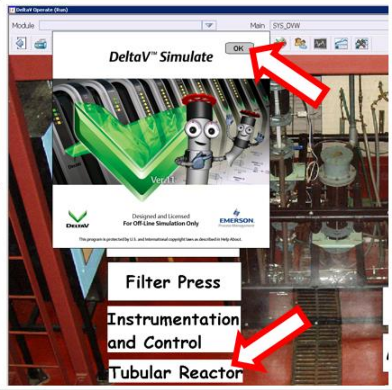
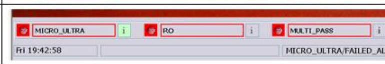
Review the SDS document for each chemical prior to conducting an experimental run with reagents. SDS documents are located in an online database; the link is provided in the CHE Laboratory page on Moodle.

Refer to the Risk Analysis document for details.

DELTAV LOGON

Step	Action and Rationale	Image
1	Turn on the Remote Operator station CHELAB-8 next to the experimental setup. **Note:** Normally the computer will be on, so only the monitor will need to be turned on.	
2	Log on to the computer: • Username: .\che (default) • Password: student	
3	Double-click the "Remote Desktop …" icon on the desktop.	

4	Connect to the default IP address (192.168.1.201): • Username: reactor (default) • Password: tubularreactor	
5	Wait for approximately 30 seconds, until DeltaV initializes.	
6	When the FlexLock ribbon appears, click on the "DeltaV Operate" button.	

7	When the DeltaV Simulate window appears, select "OK" to acknowledge the license agreement.	
8	Click on the "Tubular Reactor" button on the DeltaV main screen.	
9	Ignore any alarms that may be flashing at the bottom of the screen.	

GRAPHICAL USER INTERFACE (GUI)

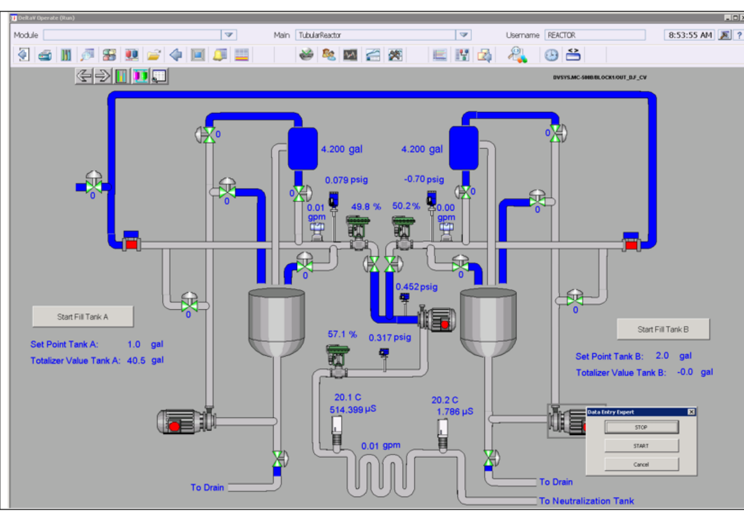
1. Experiment Main Display

This is the GUI for the Tubular Reactor.

If the process variables are not displayed when you open the file (i.e., the values appear as *** or @@@), do not proceed—call Mr. Cunning or your lab instructor.

To start or stop a pump, click on the equipment image and then select the appropriate option in the pop-up window, as shown.

When a pump is running, the red dot on the equipment image will turn green.



2. Controller Faceplates

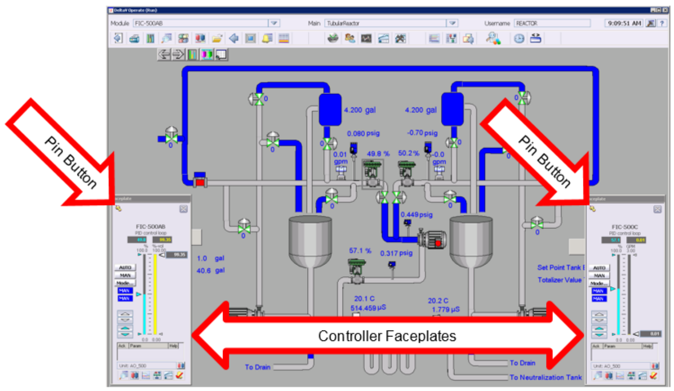
The Controller Faceplate shows and allows access to the most important operating parameters of the control algorithm.

Note: When in use, the Controller Faceplates will block portions of the GUI. Therefore it is important not to access them until instructed in the SOP.

To display the Faceplate of the mixing ratio controller (FIC-500AB), click on the equipment image for either of the ratio control valves (FIC-500A or FIC-500B). Drag the Controller Faceplate to the bottom left corner of the GUI, as shown below, and click the Pin button.

To display the Faceplate of the reactor flowrate controller (FIC-500C), click on the equipment image of the flow control valve (FIC-500C). Drag the Controller Faceplate to the bottom right corner of the GUI, as show below, and click the Pin button.

Note: Failure to click the Pin button on each Controller Faceplate will result in inability to view both Faceplates simultaneously.



2.1. FIC-500AB - Mixing Ratio Controller

The callouts in the print screen describe some of the most important fields in the Mixing Ratio Controller Faceplate (FIC-500AB).

The purpose of the mixing ratio controller is to maintain the 50/50 % vol ratio between the reagents during the reaction.

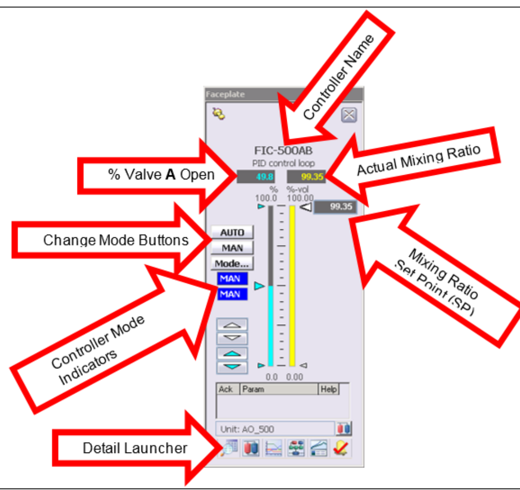
The mixing ratio controller (FIC-500AB) manipulates both control valves, FIC-500A and FIC-500B. The percent opening displayed on the controller faceplate refers to FIC-500A control valve. The percent opening of the other control valve, FIC-500B, is the balance to 100%, that is,

(% FIC-500A) + (% FIC-500B) = 100%.

To check the tuning parameters of the controller click on the Detail launcher.

The settings for the Mixing Ratio Controller Faceplate (FIC-500AB) should be:

- Gain = 0.25
- Rate = 0.0 s
- Reset = 2.26 s (Displayed as 2.3)



2.2. FIC-500C – Reactor Flowrate Controller

The callouts in the print screen describe some of the most important fields in the Reactor Flowrate Controller Faceplate (FIC-500C).

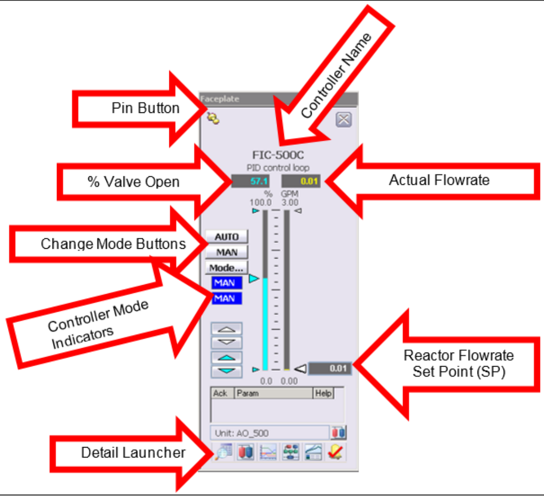
The purpose of the reactor flowrate controller is to maintain a constant flowrate through the reactor by manipulating the control valve FIC-500C.

If the opening of the control valve is less than 15%, a safety interlock will be activated that will cut the power to the reactor flow pump P-500C.

To check the tuning settings of the controller click on the Detail launcher.

The settings for the Reactor Flowrate Controller (FIC-500C) should be:

- Gain = 0.60
- Rate = 0.0 s
- Reset = 3.4 s

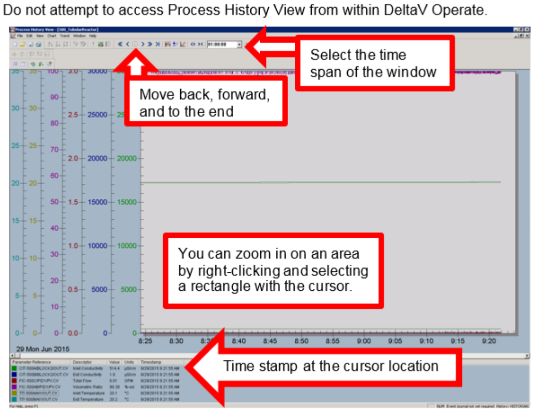


3. Process History View (PHV) Application

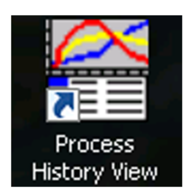
The Process History View (PHV) is an application that provides real-time and historical data in a chart recorder graphical format. It is used to monitor the progress of the experiment. It is also used to determine the start and end time in the process data retrieval procedure.

To access the Process History View utility, switch to Desktop view and double-click on the Process History View icon (1). The Process History View for the tubular reactor will load automatically. If it does not, go to "File" and select "500_TubularReactor.phvc" from the pane with recently accessed files (2).

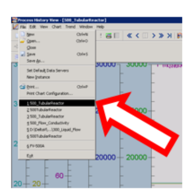
Note: Do not attempt to access Process History View from within DeltaV Operate.



(1)



(2)

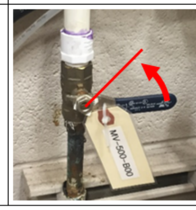


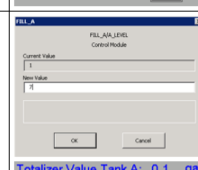
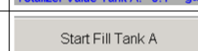
Clicking on the name of a variable at the bottom of the screen will bold the plotted line and make it easier to track.

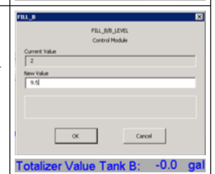
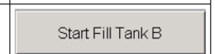
PROCEDURAL DESCRIPTIONS FOR PRACTICE RUNS WITH WATER (NO REAGENTS)

1. Equipment Preparation for a Practice Run

1.1. Filling the Storage Tanks with Water

Step	Action and Rationale	Image
1	On the P&ID, follow the "Fill with city or DI water" pathway.	
2	Check that all valves are closed.	
3	Open these manual valves: • MV-500-B05 • MV-500-B06	
4	Open the valve delivering city water (MV-500-B00) to a position halfway between open and closed. This will prevent the flow meters from exceeding their range. The maximum flow rate the flow meters can measure is 8 gpm.	

5	From the operator screen in DeltaV, click on the Set Point Tank A number. A data entry screen will pop up.	
6	Enter the set point for TK-500A as 5 gallons. **Note:** You will be making 6 gallons of EtAc solution. However, due to the time it takes for the automated valve to completely close and stop the flow, the actual volume of water filling the tank (Totalizer Value) will always be greater than the set point. We use a set point of 5 gallons to account for this. **Note:** TK-500A has a capacity of 15 gallons.	
7	Click "Start Fill Tank A" to begin filling the tank with city water.	
8	Water supply valve FV-500A will be opened automatically to start the filling process. Monitor the flowrate from the GUI or the FIT-500A flowmeter display to ensure it is less than 8 gpm. If the displayed flowrate is 8 gpm, the range of the flowmeter has been exceeded and the actual flowrate cannot be measured. In this case, • Adjust MV-500-B00 so that the displayed flowrate is less than 8 gpm • Abort the tank fill • Empty the tank • Start the tank fill procedure from the beginning	
9	The FV-500A valve will be automatically closed when the specified volume has been metered through flowmeter FIT-500A. Record the "Totalizer Value Tank A" when the filling process is complete.	

10	From the operator screen in DeltaV, click on the Set Point Tank B number. A data entry screen will pop up.	
11	Enter the Set Point for TK-500B as 7.5 gallons. **Note:** You will be making 8.5 gallons of NaOH solution. However, due to the time it takes for the automated valve to completely close and stop the flow, the actual volume of water filling the tank (Totalizer Value) will always be greater than the set point. We use a set point of 7.5 gallons to account for this. **Note:** TK-500B has a capacity of 55 gallons.	
12	Click "Start Fill Tank B" to begin filling the tank with city water.	
13	Water supply valve FV-500B will be opened automatically to start the filling process. Monitor the flowrate from the GUI or the FIT-500B flowmeter display to ensure it is less than 8 gpm. If the displayed flowrate is 8 gpm, the range of the flowmeter has been exceeded and the actual flowrate cannot be measured. In this case • Adjust MV-500-B00 so that the displayed flowrate is less than 8 gpm • Abort the tank fill • Empty the tank • Start the tank fill procedure from the beginning.	
14	The valve FV-500B will be automatically closed when the specified volume has been metered through flowmeter FIT-500B. Record the "Totalizer Value Tank B" when the filling process is complete.	Totalizer Value Tank B: -0.0 gal

15	Close these manual valves: • MV-500-B00 • MV-500-B05 • MV-500-B06	
16	**Emergency Shutdown:** In case there is a need for immediate shutoff—that is, in case of potential overflow—press the Abort Fill button for both automated valves in DeltaV. Note that the button only appears after a fill operation has started. Alternatively, close manual valve MV-500-B00.	

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1.2. Reagent Preparation

Step	Action and Rationale	Image
17	Speak to Mr. Cunning to obtain: • Metal containers in which to prepare the reagents • The location of the laboratory hood in which to prepare the reagents	
18	Calculate the volume of water that needs to be added to TK-500B in order to get 8.5 gallons. To do this, you need to subtract the Totalizer Value Tank B from 8.5 gallons.	
19	In a metal container, add this volume of tap water.	
20	Calculate the mass of solid NaOH required to make 8.5 gallons of 0.08 M solution. **Note:** If the amount you calculate is greater than 115 g, have your instructor check your calculations.	
21	At this point in the experimental run you will weigh the required amount of solid NaOH and add it to the metal container, stirring until dissolved completely.	
22	Pour the prepared solution into the NaOH storage tank, TK-500B.	
23	Calculate the volume of water that needs to be added to TK-500A in order to get 6 gallons. To do this, you need to subtract the Totalizer Value Tank A from 6 gallons.	
24	In a metal container, add this volume of tap water.	
25	Calculate the mass of 95% EtAc stock solution required to make 6 gallons of 0.1 M solution. **Note:** If the amount you calculate is greater than 245 g, have your instructor check your calculations.	
26	At this point in the experimental run you will weigh the required amount of 95% EtAc stock and add it to the metal container, stirring until fully homogeneous.	
27	Pour the prepared solution into the EtAc storage tank, TK-500A.	

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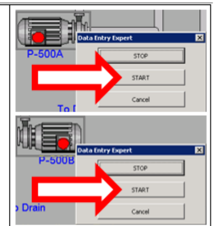
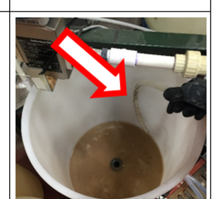
2. Conducting a Practice Run

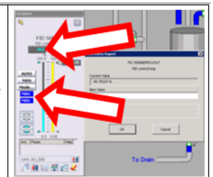
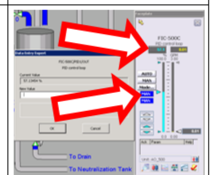
2.1. Mixing and Recirculating

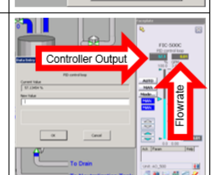
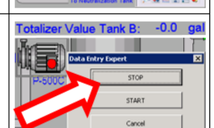
Step	Action and Rationale	Image
28	On the P&ID, follow the "Recirculation" pathway.	
29	Turn on the mechanical switch labeled "PUMPS" located on the wall to the right of the unit.	
30	Open these manual valves: • MV-500-B01 • MV-500-B02	

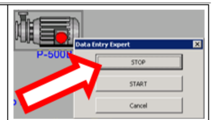
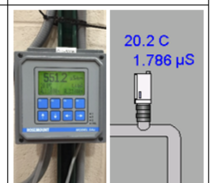
31	Turn on pumps P-500A and P-500B from the GUI. This will circulate the fluid from the storage tanks (TK-500A and TK-500B) to the holding tanks (TK-500C and TK-500D) and back to the storage tanks (via the overflow line), using the pumps as mixing devices.	
32	Wait 5 minutes to ensure good mixing.	

2.2. Conductivity of NaOH / Preloading the Neutralization Tank

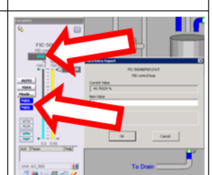
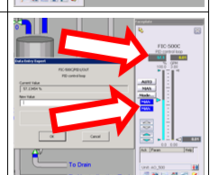
Step	Action and Rationale	Image
33	**Note:** During the experimental run, you will take a sample of the prepared NaOH solution and have it analyzed (titrated) in the Chemistry Department to find out the actual molar concentration. Make these arrangements in advance, using the information in the Experimental Progression section of the SOP.	
34	Obtain a 50 mL plastic bottle for the NaOH sample and label it with the following information: • "NaOH solution ~0.08M" • Name of the student responsible for the sample handling • Group number • Date	
35	Remove the hose from the lid of the neutralization tank. Put the lid aside. Lower the hose close to the bottom of the tank to avoid splashing during the preloading. When fluid is flowing, one person must always hold the hose in position to prevent spills. The person holding the hose must wear gloves.	
36	Display and pin the Controller Faceplates for FIC-500AB and FIC-500C to the GUI, as shown in the GUI section of the SOP.	
37	Check the tuning parameters of FIC-500AB and FIC-500C, as explained in the GUI section of the SOP.	

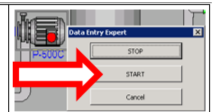
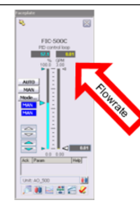
38	Verify that the mixing ratio controller FIC-500AB is in manual (MAN) mode. If not, set to MAN. Set the controller output (valve FIC-500A opening) to 0% To change the controller output, click on the teal number and entering the new value in the data entry box. This action will completely close valve FIC-500A while completely opening valve FIC-500B. Recall that in the control algorithm the openings of the two ratio control valves must add up to 100%.	
39	Verify that the reactor flow controller FIC-500C is in manual (MAN) mode. If not, set to MAN. Set the controller output (valve FIC-500C opening) to 57%.	
40	Record the current totalizer value for Tank B, displayed on the GUI.	Totalizer Value Tank B: -0.0 gal
41	Open these manual valves: • MV-500-B04 • MV-500-B10 This will start the flow of NaOH from the holding tank TK-500D through the reactor and into the neutralization tank. The neutralization tank has to be preloaded with NaOH solution in order to neutralize the unreacted EtAc during the reaction run. Recall that during the reaction run EtAc is in excess (NaOH is the limiting reagent).	

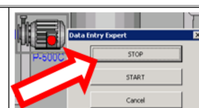
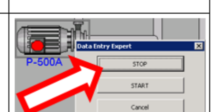
42	Turn on pump P-500C from the GUI. This will significantly increase the flowrate. **Note:** PT-500A and PT-500B must both be above 2 psig. If they are not, the pump will not start even though the light turns green on the GUI. If this happens, call your instructor immediately. Do not proceed.	
43	Once pump P-500C is on, watch the value of the flowrate (in yellow) on the flow controller faceplate. The flowrate should be between 0.8 gpm and 1.2 gpm. If the flowrate is outside of this range, change the controller output (FIC-500C opening) to bring the flowrate within this range. Record the controller output and the flowrate when its value settles.	
44	Watch the totalizer value for Tank B. When the value has increased by 4.1 gallons from the value recorded earlier, stop pump P-500C from the GUI. The flow rate will decrease but will not stop. The storage tank is being drained by virtue of gravity.	
45	Change the FIC-500C controller output to bring the flow out of the hose down to a trickle.	
46	Fill and seal the bottle to be taken for analysis. The person collecting the sample must wear gloves.	
47	Watch the totalizer value for Tank B. When the value has increased by 4.3 gallons from the value recorded earlier, close these manual valves: • MV-500-B04 • MV-500-B10 This will stop the flow of NaOH solution into the neutralization tank.	

48	TK-500B will be dry now, so quickly stop the recirculation of pump P-500B from the GUI.	
49	Close valve MV-500-B02 as soon as the recirculation pump P-500B is turned off. **Note:** The pipe from the pump goes all the way to the bottom of the constant head tank. Because of this, if the valve MV-500-B02 is left open when the pump is turned off, the fluid in the constant head tank will siphon back into the mixing tank through the recirculation pump P-500B.	
50	At this point the inlet and outlet conductivity flow cells will be filled with the NaOH solution. Record the conductivity values from the displays on the conductivity transmitters or the GUI. Expect a small difference between the readings of the two conductivity probes due to a calibration baseline drift. The baseline drift will not affect the experimental results because the data analysis method uses conductivity differences, not absolute values.	
51	Put the lid back on the neutralization tank and insert the discharge hose into the tank through the hole in the lid.	

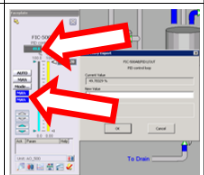
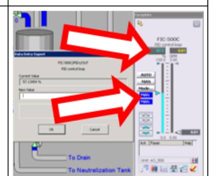
2.3. Conductivity of EtAc

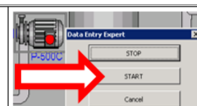
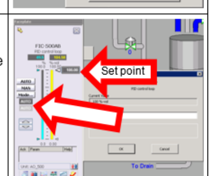
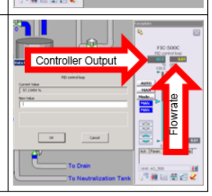
Step	Action and Rationale	Image
52	Verify that the ratio controller FIC-500AB is in manual (MAN) mode. If not, set to MAN. Set the controller output (valve FIC-500A opening) to 100%. To change the controller output, click on the teal number and entering the new value in the data entry box. This action will completely open valve FIC-500A while completely closing valve FIC-500B. Recall that in the control algorithm the openings of the two ratio control valves must add to 100%.	
53	Verify that FIC-500C controller is in manual (MAN) mode. If not, set to MAN. Set the controller output (valve FIC-500C opening) to 57%.	
54	Open these manual valves: • MV-500-B03 • MV-500-B09 This will start the flow of EtAc from the holding tank TK-500C through the reactor and into the neutralization tank.	

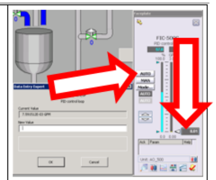
55	Turn on pump P-500C from the GUI. This will significantly increase the flowrate. **Note:** PT-500A and PT-500B must both be above 2 psig. If they are not, the pump will not start even though the light turns green on the GUI. If this happens, call your instructor immediately. Do not proceed.	
56	Once pump P-500C is on, watch the value of the flowrate. The flowrate should be between 0.8 gpm and 1.2 gpm. If the flowrate is outside of this range, change the controller output (FIC-500C opening) to bring the flowrate within this range. Record the controller output and the flowrate when its value settles.	
57	Use the display on the conductivity transmitter CIT-500A (or the GUI readings) to monitor the conductivity in the conductivity flow cell at the entrance of the reactor. In the practice run you will not see any change in the conductivity because you are using water for both reagents. In the actual experiment the conductivity will change as the EtAc solution diplaces the NaOH solution from the conductivity flow cell. **Note:** Variations in the EtAc conductivity have negligible effect on the final results because EtAc conductivity is orders of magnitude smaller than NaOH conductivity.	
58	Monitor the level of EtAc solution in the storage tank TK-500A.	

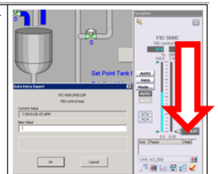
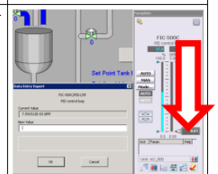
59	Stop pump P-500C from the GUI when the level in the EtAc storage tank becomes very low.	
60	Close these manual valves: • MV-500-B03 • MV-500-B09 This will stop the flow of EtAc solution into the reactor.	
61	TK-500A will be almost dry now, so quickly stop the recirculation of pump P-500A from the GUI.	
62	Close valve MV-500-B01 as soon as the pump P-500A is turned off. **Note:** The pipe from the pump goes all the way to the bottom of the constant head tank. Because of this, if the valve is left open when the pump is turned off, the fluid in the constant head tank will siphon back into the mixing tank.	
63	Record the conductivity of the solution from the display on the transmitter or the GUI.	

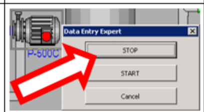
2.4. Reaction

Step	Action and Rationale	Image
64	On the P&ID, follow the "Reaction" pathway.	
65	Verify that the mixing ratio controller FIC-500AB is in manual (MAN) mode. If not, set to MAN. Set the mixing ratio controller output (valve FIC-500A opening) to 50% by clicking on the teal number and entering the new value. This action will open both valves (FIC-500A and FIC-500B) to 50% and will bring the mixing ratio between the reagents close to 50/50 by volume. The ratio is not exactly 50/50 due to differences in the piping network resistance for the two reagents between the corresponding holding tank and the mixing point.	
66	Open these manual valves simultaneously: • MV-500-B03 • MV-500-B04	
67	Verify that the flow controller FIC-500C is in manual (MAN) mode. If not, set to MAN. Set the controller output (valve FIC-500C opening) to 57%.	
68	Open these manual valves simultaneously: • MV-500-B09 • MV-500-B10	

69	Turn on pump P-500C from the GUI.	
70	Change the mode of the ratio controller FIC-500AB from MAN to AUTO. Change the set point to 50% by clicking on the input box (with the white arrow, on the right-hand side of the controller faceplate) and entering the new value.	
71	Turn your attention to the flow controller FIC-500C faceplate. Watch the value of the flowrate. The flowrate should be between 0.8 gpm and 1.2 gpm. If the flowrate is outside of this range, change the controller output (FIC-500C opening) to bring the flowrate within this range.	

72	Once the flowrate is in the range, change the mode of the flow controller FIC-500C from MAN to AUTO. Change the set point to 1 gpm by clicking on the input box (with the white arrow, on the right-hand side of the controller faceplate) and entering the new value.	
73	Launch the Process History View application as described in the GUI section of the SOP. Use it to monitor the key process variables.	
74	Pay attention to these control variables: • The mixing ratio must be maintained at 50/50 • The flowrate must be maintained at set point (1 gpm) If any of the control variables exhibits strong oscillations (more than 25% above and below the set point) or oscillations with increasing amplitude, abort the run: • Stop pump P-500C • Close manual valves MV-500-B03 and MV-500-B04 • Contact your instructor and explain the situation	
75	If both control variables are maintained close to the set point, wait until steady state is achieved. Steady state will be characterized with constant conductivities at the inlet and outlet of the reactor. In the practice run you will not see any change in the conductivity because you are using water for both reagents. In the actual experiment the conductivity will change as NaOH is consumed by the reaction. Make sure that sufficient time has elapsed before you can call a steady state. At a minimum, you need to wait the equivalent of the residence time (τ). It is suggested to wait at least 75% more than the residence time before making a steady-state decision. However, you need to do your own calculations and provide justification.	

76	Once steady state has been called, switch to the GUI screen and change the set point of the flow controller FIC-500C to 0.45 gpm. Keep the controller in AUTO mode.	
77	Go back to the Process History View and observe the response of the control variables to the set point change. If any of the control variables exhibits strong oscillations (more than 25% above and below the set point) or oscillations with increasing amplitude, abort the run. Contact your instructor and explain the situation.	
78	If both control variables are maintained close to the set point, wait until a new steady state is achieved.	
79	Once steady state has been called, switch to the GUI screen and change the set point of the flow controller FIC-500C to 0.25 gpm. Keep the controller in AUTO mode.	
80	Go back to the Process History View and observe the response of the control variables to the set point change. If any of the control variables exhibits strong oscillations (more than 25% above and below set point) or oscillations with increasing amplitude, abort the run. Contact your instructor and explain the situation.	

81	If both control variables are maintained close to set point, wait until a new steady state is achieved.	
82	Monitor the levels in the holding tanks. Stop the experiment when one of the holding tanks is about to become empty.	
83	Pump P-500C will automatically shut off when either PT-500A or PT-500B drops below 2 psig (in other words, when either TK-500C or TK-500D is almost empty). This is a temporary safety interlock condition. **Very Important:** Click on the “STOP” button on the GUI to permanently deenergize the pump. Verify that the green dot has changed color to red.	
84	Let all the solution drain into the neutralization tank.	
85	Open valve MV-500-B11 to empty the neutralization tank.	
86	Close these manual valves: • MV-500-B03 • MV-500-B04 • MV-500-B09 • MV-500-B10 • MV-500-B11	
87	Change the mode of both controllers from AUTO to MAN.	

3. Area Cleanup after a Practice Run

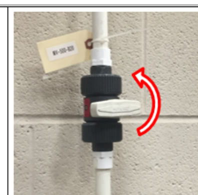
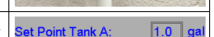
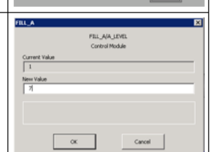
Step	Action and Rationale	Image
88	Turn off the mechanical switch labeled “PUMPS”.	
89	If this is the last practice run for the day, proceed to the DeltaV Logoff section of the SOP.	

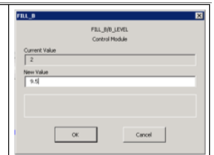
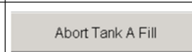
PROCEDURAL DESCRIPTIONS FOR EXPERIMENTAL RUNS WITH CHEMICAL REAGENTS

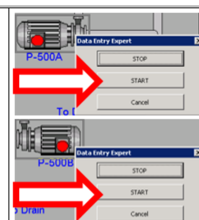
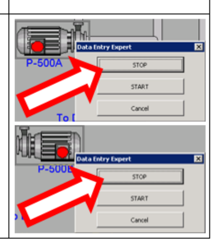
1. Equipment and Reagent Preparation for an Experimental Run with Chemicals

1.1. Flushing the System

Step	Action and Rationale	Image
1	On the P&ID, follow the "Fill with city or DI water" pathway.	
2	Check that all valves are closed.	
3	Turn on the mechanical switch labeled "PUMPS" on the wall to the right of the reactor.	
4	Open these manual valves: • MV-500-B05 • MV-500-B06	

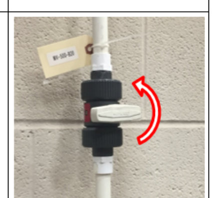
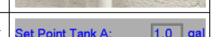
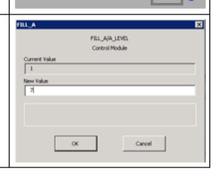
5	Open the valve delivering DI water (MV-500-B20).	
6	From the operator screen in DeltaV, click on the Set Point Tank A number. A data entry screen will pop up.	
7	Enter the Set Point for TK-500A as 5 gallons. **Note:** TK-500A has a capacity of 15 gallons.	
8	Click "Start Fill Tank A" to begin filling the tank with DI water.	
9	Water supply valve FV-500A will be opened automatically to start the filling process.	
10	The valve FV-500A will be automatically closed when the specified volume has been metered through flowmeter FIT-500A.	
11	From the operator screen in DeltaV, click on the Set Point Tank B number. A data entry screen will pop up.	

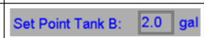
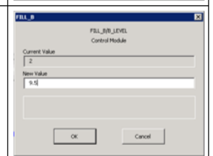
12	Assign the Set Point for TK-500B as 7.5 gallons. **Note:** TK-500B has a capacity of 55 gallons.	
13	Click "Start Fill Tank B" to begin filling the tank with DI water.	
14	Water supply valve FV-500B will be opened automatically to start the filling process.	
15	The valve FV-500B will be automatically closed when the specified volume has been metered through flowmeter FIT-500B.	
16	Close these manual valves: • MV-500-B05 • MV-500-B06 • MV-500-B20	
17	**Emergency Shutdown:** In case there is a need for immediate shutoff—that is, in case of potential overflow—press the Abort Fill button for both automated valves in DeltaV. Note that the button only appears after a fill operation has started. Alternatively, close manual valve MV-500-B20.	
18	Open these manual valves: • MV-500-B01 • MV-500-B02	

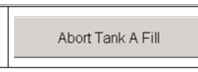
19	Turn on pumps P-500A and P-500B from the GUI. This will circulate the fluid from the storage tanks (TK-500A and TK-500B) to the holding tanks (TK-500C and TK-500D) and back to the storage tanks (via the overflow line), using the pumps as mixing devices.	
20	Wait 5 minutes to ensure good flushing.	
21	Turn off pumps P-500A and P-500B to stop recirculation.	

22	Close these manual valves: • MV-500-B01 • MV-500-B02 **Note:** The pipes from the pumps go all the way to the bottom of the constant head tanks. Because of this, if the valves are left open when the pumps are turned off, the fluid in the constant head tanks will siphon back into the mixing tanks.	
23	Open these manual valves: • MV-500-B03 • MV-500-B04 • MV-500-B09 • MV-500-B10	
24	Let all the solution drain into the neutralization tank.	
25	Open valve MV-500-B11 to empty the neutralization tank.	
26	After all tanks are empty, close these manual valves: • MV-500-B03 • MV-500-B04 • MV-500-B09 • MV-500-B10 • MV-500-B11	

1.2. Filling the Storage Tanks with Water

Step	Action and Rationale	Image
27	On the P&ID, observe the "Fill with city or DI water" pathway.	
28	Check that all valves are closed.	
29	Open these manual valves: • MV-500-B05 • MV-500-B06	
30	Open the valve delivering DI water (MV-500-B20).	
31	From the operator screen in DeltaV, click on the Set Point Tank A number. A data entry screen will pop up.	
32	Enter the Set Point for TK-500A as 5.7 gallons. **Note:** You will be making 6 gallons of EtAc solution. However, due to the time it takes for the automated valve to completely close and stop the flow, the actual volume of water filling the tank (Totalizer Value) will always be greater than the set point. We use a set point of 5 gallons to account for this. **Note:** TK-500A has a capacity of 15 gallons.	

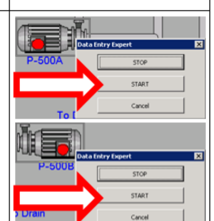
33	Click "Start Fill Tank A" to begin filling the tank with DI water.	
34	Water supply valve FV-500A will be opened automatically to start the filling process.	
35	The valve FV-500A will be automatically closed when the specified volume has been metered through flowmeter FIT-500A. Record the "Totalizer Value Tank A" when the filling process is complete.	
36	From the operator screen in DeltaV, click on the Set Point Tank B number. A data entry screen will pop up.	
37	Enter the Set Point for TK-500B as 7.5 gallons. **Note:** You will be making 8.5 gallons of NaOH solution. However, due to the time it takes for the automated valve to completely close and stop the flow, the actual volume of water (Totalizer Value) will always be greater than the set point. We use a set point of 7.5 gallons to account for this. **Note:** TK-500A has a capacity of 55 gallons.	
38	Click "Start Fill Tank B" to begin filling the tank with DI water.	
39	Water supply valve FV-500B will be opened automatically to start the filling process.	
40	The valve will be automatically closed when the specified volume has been metered through flowmeter FIT-500B. Record the "Totalizer Value Tank B" when the filling process is complete.	
41	Close these manual valves: • MV-500-B05 • MV-500-B06 • MV-500-B20	

42	**Emergency Shutdown:** In case there is a need for immediate shutoff—that is, in case of potential overflow—press the Abort Fill button for both automated valves in DeltaV. Note that the button only appears after a fill operation has started. Alternatively, close manual valve MV-500-B20.	

1.3. Reagent Preparation

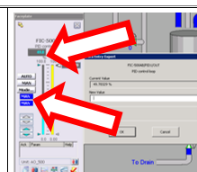
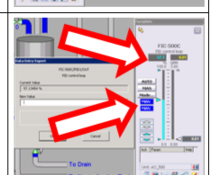
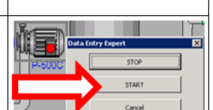
Step	Action and Rationale	Image
43	Review the appropriate SDS documents and the chemical agent release response plan for this experiment.	
44	Calculate the volume of water that needs to be added to TK-500B in order to get 8.5 gallons. To do this, you need to subtract the Totalizer Value Tank B from 8.5 gallons.	
45	In a metal container, add this volume of DI water.	
46	Weigh the appropriate amount of NaOH (as calculated during the practice run and approved by your instructor) and add it to the container.	
47	Stir the solution in the metal container until the solid dissolves.	
48	Pour the prepared solution into the NaOH storage tank, TK-500B.	
49	Calculate the volume of water that needs to be added to TK-500A in order to get 6 gallons. To do this, you need to subtract the Totalizer Value Tank A from 6 gallons.	
50	In a metal container, add this volume of DI water.	
51	Weigh the appropriate amount of 95% EtAc stock solution (as calculated during the practice run and approved by your instructor) and add it to the container.	
52	Stir the solution in the metal container thoroughly.	
53	Pour the prepared solution into the EtAc storage tank, TK-500A.	

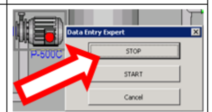
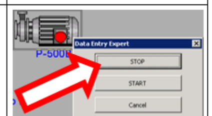
2.1. Mixing and Recirculating

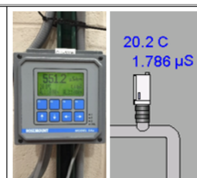
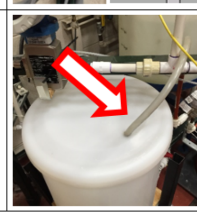
Step	Action and Rationale	Image
54	On the P&ID, observe the "Recirculation" pathway.	
55	Open these manual valves: • MV-500-B01 • MV-500-B02	
56	Turn on pumps P-500A and P-500B from the GUI. This will circulate the fluid from the storage tanks (TK-500A and TK-500B) to the holding tanks (TK-500C and TK-500D) and back to the storage tanks (via the overflow line), using the pumps as mixing devices.	
57	Wait 5 minutes to ensure good mixing.	

2.2. Conductivity of NaOH / Preloading the Neutralization Tank

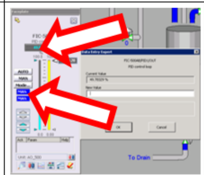
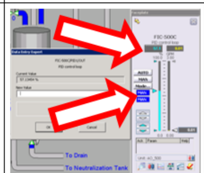
Step	Action and Rationale	Image
58	Obtain a 50 mL plastic bottle for the NaOH sample and label it with the following information: • "NaOH solution ~0.08M" • Name of the student responsible for the sample handling • Group number • Date	
59	Remove the hose from the lid of the neutralization tank. Put the lid aside. Lower the hose close to the bottom of the tank to avoid splashing during the preloading. When fluid is flowing, one person must always hold the hose in position to prevent spills. The person holding the hose must wear gloves.	
60	Display and pin the Controller Faceplates for FIC-500AB and FIC-500C to the GUI, as shown in the GUI section of the SOP.	
61	Check the tuning parameters of FIC-500AB and FIC-500C, as explained in the GUI section of the SOP.	

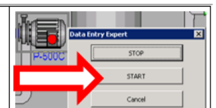
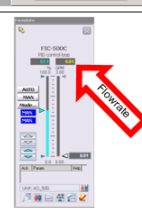
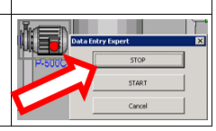
62	Verify that the ratio controller FIC-500AB is in MAN mode. If not, set to MAN. Set the controller output (valve FIC-500A opening) to 0%.	
63	Verify that the flow controller FIC-500C is in MAN mode. If not, set to MAN. Set the controller output (valve FIC-500C opening) to 57%.	
64	Record the current totalizer value for tank B, as displayed on the GUI.	Totalizer Value Tank B: -0.0 gal
65	Open these manual valves: • MV-500-B04 • MV-500-B10	
66	Turn on the pump P-500C from the GUI. **Note:** PT-500A and PT-500B must both be above 2 psig. If they are not, the pump will not start even though the light turns green on the GUI. If this happens, call your instructor immediately. Do not proceed.	

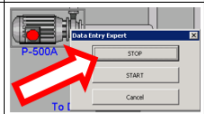
67	Once pump P-500C is on, watch the value of the flowrate (in yellow) on the flow controller faceplate. The flowrate should be between 0.8 gpm and 1.2 gpm. If the flowrate is outside of this range, change the controller output (FIC-500C opening) to bring the flowrate within this range. Record the controller output and the flowrate when its value settles.	
68	Watch the totalizer value for tank B. When the value has increased by 4.1 gallons from the value recorded earlier, stop pump P-500C from the GUI.	
69	Change the FIC-500C controller output to bring the flow out of the hose down to a trickle.	
70	Fill and seal the bottle to be taken for analysis. The person taking the sample must wear gloves.	
71	Watch the totalizer value for tank B. When the value has increased by 4.3 gallons from the value recorded earlier, close these manual valves: • MV-500-B04 • MV-500-B10	
72	TK-500B will be dry now, so quickly stop the recirculation of pump P-500B from the GUI.	
73	Close valve MV-500-B02 as soon as the pump is turned off. **Note:** The pipe from the pump goes all the way to the bottom of the constant head tank. Because of this, if the valve is left open when the pump is turned off, the fluid in the constant head tank will siphon back into the mixing tank.	

74	Record the conductivity values from the displays on the conductivity transmitters or the GUI.	
75	Put the lid back on the neutralization tank and insert the discharge hose into the tank through the hole in the lid.	

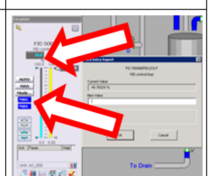
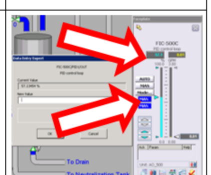
2.3. Conductivity of EtAc

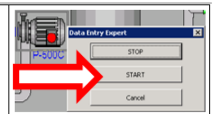
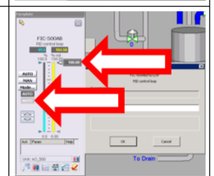
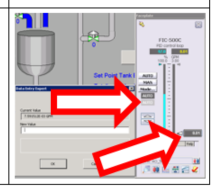
Step	Action and Rationale	Image
76	Verify that the ratio controller FIC-500AB is in MAN mode. If not, set to MAN. Set the controller output (valve FIC-500A opening) to 100%.	
77	Verify that the FIC-500C controller is in MAN mode. If not, set to MAN. Set the controller output (valve FIC-500C opening) to 57%.	
78	Open these manual valves: • MV-500-B03 • MV-500-B09	

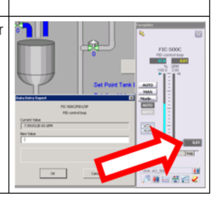
79	Turn on pump P-500C from the GUI. **Note:** PT-500A and PT-500B must both be above 2 psig. If they are not, the pump will not start even though the light turns green on the GUI. If this happens, call your instructor immediately. Do not proceed.	
80	Once pump P-500C is on, watch the value of the flowrate. The flowrate should be between 0.8 gpm and 1.2 gpm. If the flowrate is outside of this range, change the controller output (FIC-500C opening) to bring the flowrate within this range. Record the controller output and the flowrate when its value settles.	
81	Use the display on the conductivity transmitter CIT-500A (or the GUI reading) to monitor the conductivity in the conductivity flow cell at the entrance of the reactor. The conductivity will change as the EtAc solution displaces NaOH from the conductivity flow cell. **Note:** Variations in the EtAc conductivity have negligible effect on the final results because EtAc conductivity is orders of magnitude smaller than NaOH conductivity.	
82	Monitor the level of EtAc solution in the storage tank TK-500A.	
83	Stop pump P-500C from the GUI when the level in the EtAc storage tank becomes very low.	

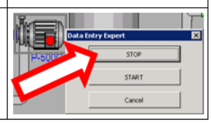
84	Close these manual valves: • MV-500-B03 • MV-500-B09	
85	TK-500A will be almost dry now, so quickly stop the recirculation of pump P-500A from the GUI.	
86	Close valve MV-500-B01 as soon as the pump is turned off. **Note:** The pipe from the pump goes all the way to the bottom of the constant head tank. Because of this, if the valve is left open when the pump is turned off, the fluid in the constant head tank will siphon back into the mixing tank.	
87	Record the conductivity of the solution from the display on the transmitter or the GUI.	

2.4. Reaction

Step	Action and Rationale	Image
88	On the P&ID, observe the "Reaction" pathway.	
89	Verify that the mixing ratio controller FIC-500AB is in MAN mode. If not, set to MAN. Set the mixing ration controller output (valve FIC-500A opening) to 50%.	
90	Open these manual valves simultaneously: • MV-500-B03 • MV-500-B04	
91	Verify that the flow controller FIC-500C is in MAN mode. If not, set to MAN. Set the controller output (valve FIC-500C opening) to 57%.	
92	Open these manual valves simultaneously: • MV-500-B09 • MV-500-B10	

93	Turn on pump P-500C from the GUI. **Note:** PT-500A and PT-500B must both be above 2 psig. If they are not, the pump will not start even though the light turns green on the GUI. If this happens, call your instructor immediately. Do not proceed.	
94	Change the mode of the ratio controller FIC-500AB from MAN to AUTO. Change the set point to 50%.	
95	Turn your attention to the flow controller FIC-500C faceplate. Watch the value of the flowrate. The flowrate should be between 0.8 gpm and 1.2 gpm. If the flowrate is outside of this range, change the controller output (FIC-500C opening) to bring the flowrate within this range.	
96	Once the flowrate is in the range, change the mode of the flow controller FIC-500C from MAN to AUTO. Change the set point to 1 gpm.	

97	Launch the Process History View application as described in the GUI section of the SOP. Use it to monitor the key process variables.	
98	Pay attention to these control variables: • The mixing ratio must be maintained at 50/50 • The flowrate must be maintained at set point (1 gpm) If any of the control variables exhibits strong oscillations (more the 25% above and below the set point) or oscillations with increasing amplitude, abort the run: • Stop pump P-500C • Close manual valves MV-500-B03 and MV-500-B04 • Contact your instructor and explain the situation	
99	If both control variables are maintained close to the set point, wait until steady state is achieved. Steady state will be characterized with constant conductivities at the inlet and outlet of the reactor. Make sure that sufficient time has elapsed before you can call a steady state. At a minimum, you need to wait the equivalent of the residence time (t). It is suggested to wait at least 75% more than the residence time before making a steady-state decision. However, you need to do your own calculations and provide justification.	
100	Once steady state has been called, switch to the GUI screen and change the set point of the flow controller FIC-500C to 0.45 gpm. Keep the controller in AUTO mode.	

101	Go back to the Process History View and observe the response of the control variables to the set point change. If any of the control variables exhibits strong oscillations (more than 25% above and below the set point) or oscillations with increasing amplitude, abort the run. Contact your instructor and explain the situation.	
102	If both control variables are maintained close to the set point, wait until a new steady state is achieved.	
103	Once steady state has been called, switch to the GUI screen and change the set point of the flow controller FIC-500C to 0.25 gpm. Keep the controller in AUTO mode.	
104	Go back to the Process History View and observe the response of the control variables to the set point change. If any of the control variables exhibits strong oscillations (more than 25% above and below the set point) or oscillations with increasing amplitude, abort the run. Contact your instructor and explain the situation.	
105	If both control variables are maintained close to the set point, wait until a new steady state is achieved.	
106	Monitor the levels in the holding tanks. Stop the experiment when one of the holding tanks is about to become empty.	
107	Pump P-500C will automatically shut off when either PT-500A or PT-500B drops below 2 psig (in other words, when either TK-500C or TK-500D is almost empty). This is a temporary safety interlock condition. **Very Important:** Click on the “STOP” button on the GUI to permanently deenergize the pump. Verify that the green dot has changed color to red.	

108	Close these manual valves: • MV-500-B03 • MV-500-B04 • MV-500-B09 • MV-500-B10	
109	Change the mode of both controllers from AUTO to MAN.	
110	Do not drain the tanks and do not flush the system!	
111	Leave the experiment overnight. The reaction will go to completion.	
112	Arrange for a time on the next day when at least one member of the group will be able to come into lab to read the conductivity and clean the equipment.	

2.5. Conductivity of the Completed Reaction

Step	Action and Rationale	Image
113	Access the process history view and verify that the conductivity measured in the conductivity flow cells has leveled off, an indication that the limiting reagent (NaOH) has been consumed and the reaction has come to completion. Record the steady-state values from both conductivity probes. Expect a small difference between the readings of the two conductivity probes due to a calibration baseline drift. The baseline drift will not affect the experimental results because the data analysis method uses conductivity differences, not absolute values.	

2. Area Cleanup after an Experimental Run with Chemicals

Step	Action and Rationale	Image
114	After the final conductivity value has been recorded, open these manual valves: • MV-500-B03 • MV-500-B04 • MV-500-B09 • MV-500-B10 • MV-500-B11	
115	Let all the tanks drain until empty, then close these manual valves: • MV-500-B03 • MV-500-B04 • MV-500-B09 • MV-500-B10 • MV-500-B11	
116	Execute the Flush procedure using city water. The procedure can be found at the beginning of the "Equipment and Reagent Preparation for an Experimental Run with Chemicals" section. Make sure that city water is used instead of DI water.	
117	Turn off the mechanical switch labeled "PUMPS".	

DATA RETRIEVAL AND TRANSFER FROM THE DELTAV HISTORIAN

Step	Action and Rationale
1	From the Process History View, determine the start and end times for data retrieval.
2	Open the Excel file "Data Template - Tubular Reactor.xlsx" located on the Desktop.
3	Immediately save the file under a different unique name.
4	You may want to create a folder on the desktop in which to save the data during the course of the experiment. As a suggestion, the folder name could be "Group xx, Fall/Spring/Winter yyyy".
5	Enter the correct start and end times in cells I1 and K1. Note that the times must be entered in cells I1 and K1, not cells C1 and E1.
6	Select cells H1 through K1; copy and paste them into cells B1 through E1. It is important to copy and paste all the cells at the same time to ensure proper operation of the data retrieval routine.
7	Watch the progress status at the bottom of the screen. Depending on the amount of data selected for retrieval, the process may take a few minutes.
8	Scroll down to verify that all the requested data have been retrieved. If this is not the case, contact your instructor.
9	Save the file.
10	Go to the "Data - Values" worksheet and make sure that it is blank. If not, delete all the contents.
11	Go back to the "Data - Links" worksheet. Select and copy all the data in it.
12	Return to the "Data - Values" worksheet and Special Paste the data as Values. Pasting the data as Values is very important. If not done, the data will not show up when you transfer the file to your laptops.
13	Save and close the file.
14	Obtain an SD card from Mr. Cunning. You should not use personal thumb drives or SD cards to transfer the data to your laptops.

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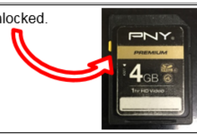
15	Verify that the card is unlocked. 
16	Copy the file from the desktop to the SD card reader. The SD card reader is Drive E under My Computer.
17	Lock the SD card before inserting it into your laptop. Locking the SD card is very important. It prevents the transfer of computer viruses from the students' laptops to the DeltaV control system. The DeltaV control system does not have virus protection.
18	Copy the file from the SD card to your laptop.
19	Unlock the card and return it to Mr. Cunning.

DELTAV LOGOFF

Proper log-off from DeltaV is very important. If you do not log off properly, you will be blocking the remote session and the group using the equipment after you may not be able to log on to the DeltaV system.

Step	Action and Rationale
1	Close DeltaV Operate (Run) by clicking the "X" in the top right corner of the computer screen.
2	Close Process History View by clicking the "X" in the top right corner of the computer screen.
3	Close any other applications.
4	Click on "Start" and select "Log Off reactor".
5	Allow the Remote Desktop application to close.
6	Click on "Start" and select "Lock".
7	Do not turn the computer off!
8	Turn the monitor off.

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REVISION HISTORY AND APPROVALS

1. Revision History

Rev.	Effective	Summary of Changes	Author(s)
1.0	29 Aug 2015	New Document	Megan Liebman
1.1	8 Feb 2016		

2. Approvals

Title	Name	Signature	Date
Responsible Faculty	Atanas Serbezov		
Laboratory Technician	Frank Cunning		
Lab Course Coordinator			
Department Head	Adam Nolte		

APPENDIX

1. Chemical Agent Release Emergency Response Plan

Because ethyl acetate is a harmful chemical, special precautions must be taken to ensure the safety of those around it. If there is an accidental release of EtAc, avoid contact with eyes or skin, and do not inhale the fumes. If a large spill occurs, quickly evacuate the lab area and alert Public Safety. For smaller spills, immediately notify Mr. Cunning, and if possible, neutralize the spill with available NaOH solution, and then flush any EtAc down the floor drains with water.

Because the concentration of the NaOH solution used in this project is so low, spills involving only NaOH are much less dangerous. For spills of NaOH, avoid contact with skin and eyes, and wash any NaOH down the floor drains with water. For larger spills, also notify Mr. Cunning.

STANDARD OPERATING PROCEDURE CHECKLIST – PRACTICE RUN

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PURPOSE

The purpose of this document is to:

- Provide a concise list of instructions that can be easily understood and followed by students who are already familiar with the experiment.
- Ensure that all experimental steps are completed and executed in the correct sequence.
- Provide accountability for the correct execution of the experiment.
- Document the operating conditions.
- Create a permanent record for the execution of the experiment.

Refer to the full SOP document for details and explanations on the individual steps.

GENERAL INSTRUCTIONS

- Print a hardcopy of this document and complete the signatures section prior to the start of the experiment.
- Have a copy of the full SOP document readily available.
- If you are running the experiment for the first time, follow the full SOP document.
- As soon as a step is completed, the person responsible for the execution of the step must initial to indicate completion.
- Initial each step as you go through the procedure.
- Upon completion of the experiment, file the document in the appropriate folder.
- A separate checklist is required for each experimental run.

SIGNATURES

Academic Year: ________________ Group Number: ______________________________

Quarter: ______________________ Instructor: __________________________________

Student Name	Signature	Initials	Date

SAFETY PRECAUTIONS

Chemical Hazard: Ethyl acetate (EtAc) is a flammable liquid and vapor. It will cause eye and skin irritation upon contact.

Chemical Hazard: Sodium hydroxide (NaOH) is a corrosive solid and liquid. It will cause eye and skin burns upon contact.

Environmental Hazard: Review the chemical agent release response plan document in the full SOP document.

Review the SDS document for each chemical prior to conducting an experimental run with reagents. SDS documents are located in an online database; the link is provided in the CHE Laboratory page on Moodle.

Refer to the Risk Analysis document for details.

DELTAV LOGON

Step	Action	Relevant Data and Units	Operator Initials
1	Turn on the Remote Operator station.		
2	Log on to the computer.		
3	Open the Remote Desktop application.		
4	Connect to the default IP address.		
5	Wait until DeltaV initializes.		
6	Launch DeltaV Operate.		
7	Acknowledge the license agreement.		
8	Open the Tubular Reactor operating station.		
9	Ignore flashing alarms.		

PROCEDURAL DESCRIPTIONS FOR PRACTICE RUNS WITH WATER (NO REAGENTS)

1. Equipment Preparation for a Practice Run

1.1. Filling the Storage Tanks with Water

Step	Action	Relevant Data and Units	Operator Initials
1	Observe the "Fill with city or DI water" pathway on the P&ID.		
2	Check that all valves are closed.		
3	Open these valves: • MV-500-B05 • MV-500-B06		
4	Open MV-500-B00 halfway.		
5	Click on the Set Point Tank A number.		
6	Enter the set point for TK-500A as 5 gallons.		
7	Click "Start Fill Tank A".		
8	As FV-500A automatically opens, monitor the flowrate to ensure it is less than 8 gpm.		
9	When the filling process is complete, record the "Totalizer Value Tank A".	Tank A Totalizer:	
10	Click on the Set Point Tank B number.		
11	Enter the set point for TK-500B as 7.5 gallons.		
12	Click "Start Fill Tank B".		
13	As FV-500B automatically opens, monitor the flowrate to ensure it is less than 8 gpm.		
14	When the filling process is complete, record the "Totalizer Value Tank B".	Tank B Totalizer:	
15	Close these valves: • MV-500-B00 • MV-500-B05 • MV-500-B06		
16	Emergency Shutdown: If needed, press the Abort Fill button or close MV-500-B00.		

1.2. Reagent Preparation

Step	Action	Relevant Data and Units	Operator Initials
17	Obtain metal containers for reagent preparation and access to laboratory hood.		
18	Calculate the volume of water required in TK-500B.	Tank B Water to Add:	
19	In a metal container, add this volume of water.		
20	Calculate the mass of solid NaOH required.	NaOH Mass:	
21	In the metal container, add this amount of NaOH.		
22	Pour the solution into TK-500B.		
23	Calculate the volume of water required in TK-500A.	Tank A Water to Add:	
24	In a metal container, add this volume of water.		
25	Calculate the mass of 95% EtAc solution required.	EtAc Mass:	
26	In the metal container, add this amount of EtAc.		
27	Pour the solution into TK-500A.		

2. Conducting a Practice Run

2.1. Mixing and Recirculating

Step	Action	Relevant Data and Units	Operator Initials
28	Observe the “Recirculation” pathway on the P&ID.		
29	Turn on the pumps switch.		

30	Open these values: • MV-500-B01 • MV-500-B02		
31	Turn on pumps P-500A and P-500B.		
32	Wait 5 minutes.		

2.2. Conductivity of NaOH / Preloading the Neutralization Tank

Step	Action	Relevant Data and Units	Operator Initials
33	Be prepared to have a sample analyzed in the Chemistry Department during the experimental run.		
34	Obtain a plastic bottle for the NaOH sample and label it.		
35	Remove the hose from the lid of the neutralization tank and hold it close to the bottom of the tank.		
36	Pin the Controller Faceplates of FIC-500AB and FIC-500C to the GUI.		
37	Check the tuning parameters of FIC-500AB and FIC-500C.		
38	Set FIC-500AB to 0% in MAN mode.		
39	Set FIC-500C to 57% in MAN mode.		
40	Record the current totalizer value for Tank B.	Tank B Totalizer:	
41	Open these valves: • MV-500-B04 • MV-500-B10		
42	Turn on pump P-500C.		
43	Adjust FIC-500C if necessary to bring the flowrate between 0.8 gpm and 1.2 gpm. Record the controller output and the flowrate when the values settle.	FIC-500C Output: FIC-500C Flowrate:	
44	When the Tank B totalizer value has increased by 4.1 gallons from Step 40, stop pump P-500C.		

45	Change FIC-500C to bring the flow out of the hose down to a trickle.		
46	Fill and seal the bottle to be taken for analysis.		
47	When the Tank B totalizer value has increased by 4.3 gallons from Step 40, close these valves: • MV-500-B04 • MV-500-B10		
48	Quickly stop pump P-500B.		
49	Immediately close valve MV-500-B02.		
50	Record the inlet and outlet conductivity values.	NaOH Conductivity: 1) 2)	
51	Replace the lid and hose in the neutralization tank.		

2.3. Conductivity of EtAc

Step	Action	Relevant Data and Units	Operator Initials
52	Set FIC-500AB to 100% in MAN mode.		
53	Set FIC-500C to 57% in MAN mode.		
54	Open these valves: • MV-500-B03 • MV-500-B09		
55	Turn on pump P-500C.		
56	Adjust FIC-500C if necessary to bring the flowrate between 0.8 gpm and 1.2 gpm. Record the controller output and the flowrate when the values settle.	FIC-500C Output: FIC-500C Flowrate:	
57	Monitor the conductivity at the entrance of the reactor.		
58	Monitor the level of EtAc solution in TK-500A.		
59	Stop pump P-500C when the level in TK-500A becomes very low.		

60	Close these valves: • MV-500-B03 • MV-500-B09		
61	Quickly stop pump P-500A.		
62	Immediately close valve MV-500-B01.		
63	Record the inlet conductivity value.	EtAc Conductivity:	

2.4. Reaction

Step	Action	Relevant Data and Units	Operator Initials
64	Observe the "Reaction" pathway on the P&ID.		
65	Set FIC-500AB to 50% in MAN mode.		
66	Open these valves simultaneously: • MV-500-B03 • MV-500-B04		
67	Set FIC-500C to 57% in MAN mode.		
68	Open these valves simultaneously: • MV-500-B09 • MV-500-B10		
69	Turn on pump P-500C.		
70	Set FIC-500AB to 50% in AUTO mode.		
71	Adjust FIC-500C if necessary to bring the flowrate between 0.8 gpm and 1.2 gpm.		
72	Set FIC-500C to 1 gpm in AUTO mode.		
73	Launch the Process History View application.		
74	Monitor the control variables. If any of them exhibit strong or increasing oscillations, abort the run.		
75	Wait until steady state is achieved.	Length of Time:	
76	Set FIC-500C to 0.45 gpm in AUTO mode.		
77	Monitor the control variables. If any of them exhibit strong or increasing oscillations, abort the run.		

78	Wait until steady state is achieved.	Length of Time:	
79	Set FIC-500C to 0.25 gpm in AUTO mode.		
80	Monitor the control variables. If any of them exhibit strong or increasing oscillations, abort the run.		
81	Wait until steady state is achieved.	Length of Time:	
82	Stop the experiment when one of the holding tanks is about to become empty.		
83	Click the “STOP” button for P-500C after it has automatically shut off.		
84	Let all the solution drain into the neutralization tank.		
85	Open MV-500-B11.		
86	After all tanks are empty, close these values: • MV-500-B03 • MV-500-B04 • MV-500-B09 • MV-500-B10 • MV-500-B11		
87	Change both controllers from AUTO to MAN.		

3. Area Cleanup after a Practice Run

Step	Action	Relevant Data and Units	Operator Initials
88	Turn off the pumps switch.		
89	If this is the last run of the day, proceed to DeltaV Logoff.		

DELTAV LOGOFF

Step	Action	Relevant Data and Units	Operator Initials
1	Close DeltaV Operate (Run).		
2	Close Process History View.		
3	Close any other applications.		
4	Log off from DeltaV.		
5	Allow the Remote Desktop application to close.		
6	Lock the computer.		
7	Do not turn the computer off!		
8	Turn the monitor off.		

STANDARD OPERATING PROCEDURE CHECKLIST – RUN WITH CHEMICALS

Area 500 – Tubular Reactor	Revision 1.1 Effective 8 February 2016	Page 1 of 12

PURPOSE

The purpose of this document is to:

- Provide a concise list of instructions that can be easily understood and followed by students who are already familiar with the experiment.
- Ensure that all experimental steps are completed and executed in the correct sequence.
- Provide accountability for the correct execution of the experiment.
- Document the operating conditions.
- Create a permanent record for the execution of the experiment.

Refer to the full SOP document for details and explanations on the individual steps.

GENERAL INSTRUCTIONS

- Print a hardcopy of this document and complete the signatures section prior to the start of the experiment.
- Have a copy of the full SOP document readily available.
- If you are running the experiment for the first time, follow the full SOP document.
- As soon as a step is completed, the person responsible for the execution of the step must initial to indicate completion.
- Initial each step as you go through the procedure.
- Upon completion of the experiment, file the document in the appropriate folder.
- A separate checklist is required for each experimental run.

SIGNATURES

Academic Year: ______________ Group Number: ________________________________

Quarter: ____________________ Instructor: ____________________________________

We, the undersigned, have read the SDS documents for ethyl acetate and sodium hydroxide and understood the information in them.

Student Name	Signature	Initials	Date

SAFETY PRECAUTIONS

Chemical Hazard: Ethyl acetate (EtAc) is a flammable liquid and vapor. It will cause eye and skin irritation upon contact.

Chemical Hazard: Sodium hydroxide (NaOH) is a corrosive solid and liquid. It will cause eye and skin burns upon contact.

Environmental Hazard: Review the chemical agent release response plan document in the full SOP document.

Review the SDS document for each chemical prior to conducting an experimental run with reagents. SDS documents are located in an online database; the link is provided in the CHE Laboratory page on Moodle.

Refer to the Risk Analysis document for details.

DELTAV LOGON

Step	Action	Relevant Data and Units	Operator Initials
1	Turn on the Remote Operator station.		
2	Log on to the computer.		
3	Open the Remote Desktop application.		
4	Connect to the default IP address.		
5	Wait until DeltaV initializes.		
6	Launch DeltaV Operate.		
7	Acknowledge the license agreement.		
8	Open the Tubular Reactor operating station.		
9	Ignore flashing alarms.		

PROCEDURAL DESCRIPTIONS FOR EXPERIMENTAL RUNS WITH CHEMICAL REAGENTS

1. Equipment and Reagent Preparation for an Experimental Run with Chemicals

1.1. Flushing the System

Step	Action	Relevant Data and Units	Operator Initials
1	Observe the "Fill with city or DI water" pathway on the P&ID.		
2	Check that all valves are closed.		
3	Turn on the pumps switch.		
4	Open these valves: • MV-500-B05 • MV-500-B06		
5	Open MV-500-B20.		
6	Click on the Set Point Tank A number.		
7	Enter the set point for TK-500A as 5 gallons.		
8	Click "Start Fill Tank A".		
9	As FV-500A automatically opens, monitor the flowrate to ensure it is less than 8 gpm.		
10	FV-500A will close automatically.		
11	Click on the Set Point Tank B number.		
12	Enter the set point for TK-500B as 7.5 gallons.		
13	Click "Start Fill Tank B".		
14	As FV-500B automatically opens, monitor the flowrate to ensure it is less than 8 gpm.		
15	FV-500B will close automatically.		
16	Close these valves: • MV-500-B05 • MV-500-B06 • MV-500-B20		
17	Emergency Shutdown: If needed, press the Abort Fill button or close MV-500-B20.		
18	Open these valves: • MV-500-B01 • MV-500-B02		
19	Turn on pumps P-500A and P-500B.		

20	Wait 5 minutes.		
21	Turn off pumps P-500A and P-500B.		
22	Close these valves: • MV-500-B01 • MV-500-B02		
23	Open these valves: • MV-500-B03 • MV-500-B04 • MV-500-B09 • MV-500-B10		
24	Let all the solution drain into the neutralization tank.		
25	Open MV-500-B11.		
26	After all tanks are empty, close these valves: • MV-500-B03 • MV-500-B04 • MV-500-B09 • MV-500-B10 • MV-500-B11		

1.2. Filling the Storage Tanks with Water

Step	Action	Relevant Data and Units	Operator Initials
27	Observe the "Fill with city or DI water" pathway on the P&ID.		
28	Check that all valves are closed.		
29	Open these valves: • MV-500-B05 • MV-500-B06		
30	Open MV-500-B20.		
31	Click on the Set Point Tank A number.		
32	Enter the set point for TK-500A as 5 gallons.		
33	Click "Start Fill Tank A".		
34	As FV-500A automatically opens, monitor the flowrate to ensure it is less than 8 gpm.		
35	When the filling process is complete, record the "Totalizer Value Tank A".	Tank A Totalizer:	

36	Click on the Set Point Tank B number.		
37	Enter the set point for TK-500B as 7.5 gallons.		
38	Click "Start Fill Tank B".		
39	As FV-500B automatically opens, monitor the flowrate to ensure it is less than 8 gpm.		
40	When the filling process is complete, record the "Totalizer Value Tank B".	Tank B Totalizer:	
41	Close these valves: • MV-500-B05 • MV-500-B06 • MV-500-B20		
42	Emergency Shutdown: If needed, press the Abort Fill button or close MV-500-B20.		

1.3. Reagent Preparation

Step	Action	Relevant Data and Units	Operator Initials
43	Review the SDS documents and chemical agent release response plan associated with this experiment. **All operators must complete and initial this step.**		
44	Calculate the volume of water required in TK-500B.	Tank B Water to Add:	
45	In a metal container, add this volume of DI water.		
46	Weigh the appropriate amount of NaOH and add it to the container.	NaOH Mass:	
47	Stir the solution in the container until the solid dissolves.		
48	Pour the solution into TK-500B.		
49	Calculate the volume of water required in TK-500A.	Tank A Water to Add:	
50	In a metal container, add this volume of DI water.		

51	Weigh the appropriate amount of NaOH and add it to the container.	EtAc Mass:	
52	Stir the solution in the container thoroughly.		
53	Pour the solution into TK-500A.		

2. Conducting an Experimental Run with Chemicals

2.1. Mixing and Recirculating

Step	Action	Relevant Data and Units	Operator Initials
54	Observe the "Recirculation" pathway on the P&ID.		
55	Open these valves: • MV-500-B01 • MV-500-B02		
56	Turn on pumps P-500A and P-500B.		
57	Wait 5 minutes.		

2.2. Conductivity of NaOH / Preloading the Neutralization Tank

Step	Action	Relevant Data and Units	Operator Initials
58	Obtain a plastic bottle for the NaOH sample and label it.		
59	Remove the hose from the lid of the neutralization tank and hold it close to the bottom of the tank.		
60	Pin the Controller Faceplates of FIC-500AB and FIC-500C to the GUI.		
61	Check the tuning parameters of FIC-500AB and FIC-500C.		
62	Set FIC-500AB to 0% in MAN mode.		
63	Set FIC-500C to 57% in MAN mode.		
64	Record the current totalizer value for Tank B.	Tank B Totalizer:	

65	Open these valves: • MV-500-B04 • MV-500-B10		
66	Turn on pump P-500C.		
67	Adjust FIC-500C if necessary to bring the flowrate between 0.8 gpm and 1.2 gpm. Record the controller output and the flowrate when the values settle.	FIC-500C Output: FIC-500C Flowrate:	
68	When the Tank B totalizer value has increased by 4.1 gallons from Step 64, stop pump P-500C.		
69	Change FIC-500C to bring the flow out of the hose down to a trickle.		
70	Fill and seal the bottle to be taken for analysis.		
71	When the Tank B totalizer value has increased by 4.3 gallons from Step 64, close these valves: • MV-500-B04 • MV-500-B10		
72	Quickly stop pump P-500B.		
73	Immediately close valve MV-500-B02.		
74	Record the inlet and outlet conductivity values.	NaOH Conductivity: 1) 2)	
75	Replace the lid and hose in the neutralization tank.		

2.3. Conductivity of EtAc

Step	Action	Relevant Data and Units	Operator Initials
76	Set FIC-500AB to 100% in MAN mode.		
77	Set FIC-500C to 57% in MAN mode.		
78	Open these valves: • MV-500-B03 • MV-500-B09		

79	Turn on pump P-500C.		
80	Adjust FIC-500C if necessary to bring the flowrate between 0.8 gpm and 1.2 gpm. Record the controller output and the flowrate when the values settle.	FIC-500C Output: FIC-500C Flowrate:	
81	Monitor the conductivity at the entrance of the reactor.		
82	Monitor the level of EtAc solution in TK-500A.		
83	Stop pump P-500C when the level in TK-500A becomes very low.		
84	Close these valves: • MV-500-B03 • MV-500-B09		
85	Quickly stop pump P-500A.		
86	Immediately close valve MV-500-B01.		
87	Record the inlet conductivity value.	EtAc Conductivity:	

2.4. Reaction

Step	Action	Relevant Data and Units	Operator Initials
88	Observe the “Reaction” pathway on the P&ID.		
89	Set FIC-500AB to 50% in MAN mode.		
90	Open these valves simultaneously: • MV-500-B03 • MV-500-B04		
91	Set FIC-500C to 57% in MAN mode.		
92	Open these valves simultaneously: • MV-500-B09 • MV-500-B10		
93	Turn on pump P-500C.		
94	Set FIC-500AB to 50% in AUTO mode.		

95	Adjust FIC-500C if necessary to bring the flowrate between 0.8 gpm and 1.2 gpm.		
96	Set FIC-500C to 1 gpm in AUTO mode.		
97	Launch the Process History View application.		
98	Monitor the control variables. If any of them exhibit strong or increasing oscillations, abort the run.		
99	Wait until steady state is achieved.	Length of Time:	
100	Set FIC-500C to 0.45 gpm in AUTO mode.		
101	Monitor the control variables. If any of them exhibit strong or increasing oscillations, abort the run.		
102	Wait until steady state is achieved.	Length of Time:	
103	Set FIC-500C to 0.25 gpm in AUTO mode.		
104	Monitor the control variables. If any of them exhibit strong or increasing oscillations, abort the run.		
105	Wait until steady state is achieved.	Length of Time:	
106	Stop the experiment when one of the holding tanks is about to become empty.		
107	Click the “STOP” button for P-500C after it has automatically shut off.		
108	Close these valves: • MV-500-B03 • MV-500-B04 • MV-500-B09 • MV-500-B10		
109	Change both controllers from AUTO to MAN.		
110	Do not drain the tanks and do not flush the system!		
111	Leave the experiment overnight.		

112	Arrange for a time on the next day when at least one member of the group will be able to come into lab to read the conductivity and clean the equipment.	Date: Time: Group Member(s):	

2.5. Conductivity of the Completed Reaction

Step	Action	Relevant Data and Units	Operator Initials
113	Read the values from the conductivity transmitters.	100% Conductivity: 1) 2)	

3. Area Cleanup after an Experimental Run with Chemicals

Step	Action	Relevant Data and Units	Operator Initials
114	After the final conductivity value has been recorded, open these valves: • MV-500-B03 • MV-500-B04 • MV-500-B09 • MV-500-B10 • MV-500-B11		
115	After all tanks are empty, close these valves: • MV-500-B03 • MV-500-B04 • MV-500-B09 • MV-500-B10 • MV-500-B11		
116	Rinse the system with city water.		
117	Turn off the pumps switch.		

DATA RETRIEVAL AND TRANSFER FROM THE DELTAV HISTORIAN

Step	Action	Relevant Data and Units	Operator Initials
1	Determine the start and end times for data retrieval.	Start Time: End Time:	
2	Open "Data Template – Tubular Reactor.xlsx".		
3	Immediately save the file under a different unique name.	File Name:	
4	If not previously done, create a folder on the desktop.		
5	Enter the correct start and end times in cells I1 and K1.		
6	Select cells H1 through K1; copy and paste them into cells B1 through E1.		
7	Wait until the status at the bottom of the screen indicates that the transfer is complete.		
8	Verify that all requested data have been retrieved.		
9	Save the file.		
10	Clear all contents from the "Data – Values" worksheet.		
11	Select and copy all data from the "Data – Links" worksheet.		
12	Special Paste the data as Values in the "Data – Values" worksheet.		
13	Save and close the file.		
14	Obtain an SD card from Mr. Cunning.		
15	Verify that the card is unlocked.		
16	Copy the file from the desktop to the SD card reader.		
17	Lock the SD card before inserting it into your laptop.		
18	Copy the file from the SD card to your laptop.		
19	Unlock the card and return it to Mr. Cunning.		

DELTAV LOGOFF

Step	Action	Relevant Data and Units	Operator Initials
1	Close DeltaV Operate (Run).		
2	Close Process History View.		
3	Close any other applications.		
4	Log off from DeltaV.		
5	Allow the Remote Desktop application to close.		
6	Lock the computer.		
7	Do not turn the computer off!		
8	Turn the monitor off.		