I. Approvals

Signing below indicates agreement that the protocol is ready for execution of the Installation, Operational, and Performance Qualification for Crystallizer #6, located at 102 Commerce Street at the Main Plant production facility.

Project Member	Functional Area	Signature	Date
Patrick Owen	Engineering		12/20/16
Kenneth Basehore	Engineering		12/20/16
Sammy Henson	Maintenance		12/21/16
Jason Bumgarner	Production		12-21-16
Matt Haynes	Operations		12-21-16
Deborah Durbin	Quality		12-21-16

A final summary report that consists of results and conclusions based on the data collected after protocol execution will be written and approved. The executed protocol will be attached behind the report.

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II. Purpose

The purpose of this report is to provide documented evidence that Crystallizer #6 is installed properly, is operational, and functions as intended throughout its anticipated operating ranges. This will also serve as a baseline of documentation for the installation for future change control and trouble shooting. This protocol sets forth the objectives, methodology, documentation and test activities needed to complete the Installation Qualification (IQ), Operational Qualification (OQ) and Performance Qualification (PQ) for Crystallizer #6 located in the Main Plant at 102 Commerce Street in Waynesville, NC.

III. Background

1. *Historical*

Giles Chemical is a producer of Epsom Salt (magnesium sulfate) and has been producing Epsom Salt at the Waynesville, NC facility since 1950. A variety of Crystallizers have been used. In 1988 and Oslo type Crystallizer (Crystallizer #1) was installed and subsequently Crystallizer #2 (1998), #3 (2005), #4 (2013) and #5 (2015) were also installed. All of the older types of Crystallizers were removed from the facility.

2. *Current Project*

A Giles Authorization for Expenditure (AFE) was signed in 2016 to purchase and install Crystallizer #6 by the end of the 1st quarter of 2017. The vessel design was an exact copy of #2, #3, #4 and #5 Crystallizers. The machine will crystalize USP grade Epsom Salt from brine produced at Giles' manufacturing facility.

IV. Overview

Crystallizer #6 uses vacuum to cool a continuous stream of saturated brine to form crystals, which are then discharged to existing centrifuges.

V. System Description

There are six systems that make up Crystallizer #6. All are in operation when Crystallizer #6 is in operation.

1. *Tank and Circulation System*

The Crystallizer vessel holds a volume of crystal slurry and circulates it. This allows adequate time for crystal growth and selective discharge of larger crystal size distribution from fluidizing the slurry. This system consists of the main vessel, circulation pump (referred to as 'elbow pump') and circulation pump piping.

2. *Feed System*

The feed system supplies brine to the crystallizer. This system consists of the brine feed pump, brine feed flow meter, variable speed drive and pressure/level sensor.

3. *Vacuum System*

The vacuum system pulls a vacuum on the Crystallizer, allowing evaporative cooling to take place. The temperature in the vessel is controlled indirectly by the vacuum system because the vapor space approaches

thermodynamic equilibrium with the slurry and the vapor space contains only water vapor. This system consists of a barometric condenser, cooling water pump, steam ejector, ejector condenser, liquid ring vacuum pump, vacuum control valve and vacuum sensor.

4. *Fine Salt Reduction Loop*

The fine sale loop pulls smaller crystals from the upper part of the vessel, uses heat to dissolve them, and re-introduces the resultant brine into the circulation system. The system consists of fine salt loop piping, fine salt loop flow meter, fine salt loop pump, fine salt loop heat exchanger, and fine salt loop steam control valve.

5. *Discharge System*

A continuous stream of crystal slurry is pumped from the crystallizer to a centrifuge. This system consists of discharge piping, discharge pump, discharge flow meter, and discharge pump variable speed drive.

6. *Mother Liquor System*

A portion of the liquid separated from the crystals at the centrifuge is returned to the crystallizer to make up for evaporative volume loss from the cooling process. This system consists of the mother liquor flow meter and mother liquor piping. Note that an existing, common mother liquor pump will be used, so it is not included in this validation protocol.

VI. Scope

This study will be performed on Crystallizer #6. This protocol will define the test procedures, documentation, references and acceptance criteria used to establish that the system is installed properly, operates properly, and performs as expected. The executed protocol will verify that all acceptance criteria have been met, and that the crystallizer meets cGMP requirements.

VII. Roles and Responsibilities

1. Engineering
   - Write and issue the protocol
   - Investigate protocol deviation reports
   - Execute the IQ, OQ and PQ portions of the validation
   - Review the data and originate the interim notification to Quality Assurance
   - Write and route the final report
2. Quality Assurance
   - Review and approve the protocol
   - Review and approve the raw data and notifications
   - Review, approve and store the final report
3. Maintenance
   - Provide equipment manuals needed to execute the validation
   - Review and approve the protocol

- Review and approve the raw data and notifications
- Review and approve the final report

4. Production
   - Review and approve the protocol
   - Review and approve the raw data and notifications
   - Review and approve the final report
   - Assist, as needed with the execution of the IQ, OQ and PQ

VIII. Test Program

1. *Installation Qualification (IQ)*

a. Objective

The objective of the installation verification is to document that each of the 6 systems that compromise Crystallizer #6 are installed properly and document the components of each system for future reference.

b. Equipment and Materials

- Crystallizer #6
- Fluke Multimeter
- Level

c. Procedure

Perform each listed below for Crystalizer #6

1. *Location*
   Verify that the equipment is situated to allow sufficient room around the machine for Maintenance and Operations to perform their respective duties.
2. *Level*
   Verify that the instrument is level
3. *Vessel*
   Ensure that all hatches and outlets are tightened, plumbed or blanked.
4. *Plumbing*
   a. Ensure the elbow pump and plumbing is in place
   b. Ensure the brine feed pump and plumbing feeds from the brine feed tank to the elbow pump loop are in place
   c. Ensure the fine salt loop plumbing and feeds from the vessel and returns to the elbow pump loop are in place
   d. Ensure the discharge plumbing connects from the discharge outlet to the centrifuge inlet
   e. Ensure the mother liquor plumbing feeds from the mother liquor header to the elbow pump loop

f. Ensure the large condenser plumbing feeds from the creek tank to the large condenser and from the large condenser to the hot well
g. Ensure the small condenser plumbing feeds from the creek tank to the small condenser and from the small condenser to the hot well
h. Ensure city water is available to the vacuum pump
i. Ensure steam is available to the steam ejector
j. Ensure the vapor pipe connects from the top of the Crystallizer to the large condenser
k. Ensure the internal vent pipe connects from the lower part of the Crystallizer to the head of the unit

5. *Instrumentation*
   a. Ensure the discharge flow meter is in the discharge pipe
   b. Ensure the mother liquor flow meter is in the mother liquor feed pipe
   c. Ensure the brine feed flow meter is in the brine feed pipe
   d. Ensure the fine salt loop flow meter is in the fine salt loop piping
   e. Ensure the densityBrine and vacuum meters are installed on the body of the vessel
6. *Utilities*
   a. Ensure the voltage is correct to the pump drive panel
   b. Ensure the voltage is correct to the instrumentation
   c. Ensure the vacuum control valve has air pressure
   d. Ensure the fine salt loop control valve has air pressure
   e. Ensure the steam ejector has steam
   f. Ensure the fine salt loop has steam and a condensate return

d. Acceptance Criteria

If each item of the outlined procedure passes verification, then Crystallizer #6 will be installed properly.

2. *Operational Qualification (OQ)*

a. Objective

The objective of the operational qualification is to document that the components of the 6 systems that comprise Crystallizer #6 are operable and oriented correctly for the machine to operate.

b. Equipment and Materials

- Crystallizer #6

c. Procedure

Perform each listed below for Crystalizer #6

1. *Vessel and Circulation*
   a. Ensure the elbow pump is turning in the correct direction
   b. With fluid in the crystallizer, turn on the circulation and ensure a flow is coming out in the headspace by looking through the top sight glass

2. *Brine Feed System*
   a. Ensure flow meter arrow is pointing toward the elbow loop plumbing
   b. Turn on the bring pump and ensure the motor has the correct rotation
   c. Ensure the brine feed to the crystallizer shows a non-zero flow rate
3. *Vacuum System*
   a. Put the controller in manual, and cycle the valve to ensure it operates correctly
   b. With fluid in the crystallizer, turn on the vacuum system and ensure it has both water and steam present
   c. Using the vacuum gage, ensure the system pressure drops when the vacuum system is operational
4. *Discharge System*
   a. Ensure the discharge pump motor has the correct rotation
   b. With the vessel full of fluid, open the discharge valves, turn on the discharge pump, and ensure that fluid is pumped to the centrifuge
5. *Fine Salt Reduction System*
   a. Put the controller in manual, and cycle the steam valve
   b. With the vessel full of fluid, enter a fine salt loop temperature setpoint of 41 °C
   c. Verify that there is flow through the fine salt loop, and that steam is supplied when the temperature drops below the acceptable range
6. *Mother Liquor System*
   a. Ensure that the flow meter arrow is pointed toward towards the crystallizer
   b. Open the supply valve, and ensure that the flow meter shows a non-zero flow

d. Acceptance Criteria

If each item of the outlined procedure passes verification, then Crystallizer #6 will be installed properly.

3. *Performance Qualification (PQ)*

a. Objective

The objective of the performance testing is to document that Crystallizer #6 performs the functions required by Giles Chemical. Namely:

- The vacuum pulled by the vacuum system is sufficient to cool the brine solution to 35 °C
- The fines reduction loop will heat a flow of at least 15 gpm to 41 ± 3 °C
- The discharge system will pump 22 ± 3 gpm for at least 1 hour without interruption
- The plant will produce USP grade product with Crystallizer #6 runing

b. Equipment and Materials

- Crystallizer #6
- Brine
- Mother liquor
- Factory calibrated instrumentation installed on the vessel

c. Procedure

Fill and run Crystallizer #6 according to procedure for at least 24 hours before making observations. The crystallizer should be in equilibrium during the measurement collection.

1. *Vacuum System*
   Observe and record the vacuum pressures every 30 minutes for 4 hours
2. *Fines Reduction Loop*
   Observe and record the flow and temperature every 30 minutes for 4 hours
3. *Discharge System*
   Observe and record the discharge flow rate every 30 minutes for 4 hours
4. *Circulation System*
   Observe and record the circulation flow rate every 30 minutes for 4 hours
5. *Feed System*
   Observe and record the brine feed flow rate every 30 minutes for 4 hours
6. *Mother Liquor System*
   Observe and record the mother liquor flow rate every 30 minutes for 4 hours
7. *USP testing*
   Obtain USP testing results from the laboratory from before the crystallizer is running, and after the crystallizer has been running

d. Acceptance Criteria

Crystallizer #6 will be considered qualified if the IQ and OQ are completed, and all deviations have been investigated and closed out, and if each of the following PQ testing has passed:

- The absolute pressure of the vacuum system cannot exceed 1.5 inches Hg in any observation
- The fines reduction loop cannot drop below 15 gpm for any observation
- The fines reduction loop cannot fall outside of 41 ± 3 °C for any observation
- The discharge flow cannot fall outside of 22 ± 3 gpm for any observation
- Brine Feed flow cannot fall outside of 25 ± 3 gpm for any observation
- Mother liquor flow is non-zero for the duration of the observation
- Both the beginning and ending USP samples pass testing

IX. Calibration

Verify that all instruments used are within the calibration dates.

- Fluke Multimeter
- Discharge flow meter
- Brine feed flow meter
- High level pressure transmitter
- Low level pressure transmitter
- Vacuum probe
- Fines loop flow meter
- Fines loop temperature transmitter

- Mother liquor flow meter

X. References

P12-PR-200-026: Crystallizer Operations
P12-PR-200-028: Filling and Starting a Crystallizer

Installation Qualification (IQ)

1. *Equipment*

Device	Calibration Date	Calibration Expiration	Verified By	Date
Multimeter Model: Fluke 114 S/N: 36250117WS				

Expected	Actual	Pass/Fail	Verified By	Date
Crystallizer #6				
Brine Feed Flow Meter Order 53P40-EL0B1AA0BAAA S/N L80AB116000				
Discharge Flow Meter Order 53P40-EL0B1AA0BAAA S/N A5061716000				
Fines Loop Flow Meter/TT Order 83S25-1V70/0 S/N L9042816000				
Mother Liquor Flow Meter Order 53W25-UL0B1AA0BAAA S/N L904F916000				
Low Level Densitometer Order PMC71-1D390/0 S/N L904D21509C				
High Level Densitometer Order PMC71-1D390/0 S/N L904D31509C				
Vacuum Gage Order PMC71-1D382/0 S/N L903891509C				
Brine Feed Pump 2SS1G5F5				
Fine Loop Pump Goulds 3657 S/N B1601301				

Circulation Pump EP60-18-326T S/N 0516336025				
Discharge Pump Goulds 3657 S/N B1601302				
Vacuum Pump Flowserve LPHR 45317 AB AAB 0E 0 S/N CA1711194-01				

2. *Acceptance Testing*

Expected	Actual	Pass/Fail	Verified By	Date
There is sufficient room around the vessel for maintenance and operations to work	There __ sufficient room around the vessel for maintenance and operations to work			
The vessel is level	The vessel _____ level			
All hatches and outlets are tightened, plumbed or blanked	All hatches and outlets _____ tightened, plumbed or blanked			
The elbow pump is in place	The elbow pump ____ in place			
The circulation loop plumbing is in place	The circulation loop plumbing ____ in place			
The brine feed pump is in place	The brine feed pump ____ in place			
The brine feed plumbing connects the brine feed tank to the circulation loop	The brine feed plumbing _____ the brine feed tank to the circulation loop			
The fine salt loop pump is in place	The fine salt pump ____ in place			
The fine salt loop plumbing connects the vessel to the circulation loop	The fine salt loop plumbing _____ the vessel to the circulation loop			
The discharge pump is in place	The discharge pump _____ in place			

The discharge plumbing connects the discharge outlet to the centrifuge	The discharge plumbing _____ the discharge outlet to the centrifuge			
The mother liquor plumbing connects the mother liquor header to the circulation loop	The mother liquor plumbing _____ the mother liquor header to the circulation loop			
The large condenser plumbing connects the creek tank to the large condenser	The large condenser plumbing _____ the creek tank to the large condenser			
The large condenser plumbing connects the large condenser to the hot well	The large condenser plumbing _____ the large condenser to the hot well			
The small condenser plumbing connects the creek tank to the small condenser	The small condenser plumbing _____ the creek tank to the small condenser			
The small condenser plumbing connects the small condenser to the hot well	The small condenser plumbing _____ the small condenser to the hot well			
City water is available to the vacuum pump	City water _____ available to the vacuum pump			
Steam is available to the steam ejector	Steam _____ available to the steam ejector			
The vapor pipe connects the top of the crystallizer to the large condenser	The vapor pipe _____ the top of the crystallizer to the large condenser			
The internal vent pipe connects the lower part of the vessel to the vessel head	The internal vent pipe _____ the lower part of the vessel to the vessel head			
The discharge flow meter is in the discharge pipe	The discharge flow meter ____ in the discharge pipe			
The mother liquor flow meter is in the mother liquor feed pipe	The mother liquor flow meter _____ in the mother liquor feed pipe			
The brine feed flow meter is in the brine feed flow pipe	The brine feed flow meter ____ in the brine feed flow pipe			
The fine salt loop flow meter is in the fine salt loop piping	The fine salt loop flow meter _____ in the fine salt loop piping			

The density meters are both installed in the vessel	The density meters _____ both installed in the vessel			
The vacuum meter is installed in the head of the vessel	The vacuum meter ____ installed in the head of the vessel			
The voltage supplied to the pump drive panel is 460V ± 20 V				
The voltage supplied to the controls is 115V ± 10V				
The vacuum control valve has air pressure	The vacuum control valve ____ air pressure			
The fine salt loop control valve has air pressure	The fine salt loop control valve _____ air pressure			
The steam ejector has steam	The steam ejector _____ steam			
The fine salt loop has steam and a condensate return	The fine salt loop _____ steam and a condensate return			

3. *Acceptance of Testing and Review*

Expected	Actual	Pass/Fail	Verified By	Date
All actual results match the expected values.	All actual results _____ the expected values.			
Results reviewed and accepted by				

XI. Operational Qualification (OQ)

1. *Equipment*

Device	Calibration Date	Calibration Expiration	Verified By	Date
Multimeter Model: Fluke 114 S/N: 36250117WS				

Expected	Actual	Pass/Fail	Verified By	Date
Crystallizer #6				
Brine Feed Flow Meter Order 53P40-EL0B1AA0BAAA S/N L80AB116000				
Discharge Flow Meter Order 53P40-EL0B1AA0BAAA S/N A5061716000				
Fines Loop Flow Meter/TT Order 83S25-1V70/0 S/N L9042816000				
Mother Liquor Flow Meter Order 53W25-UL0B1AA0BAAA S/N L904F916000				
Low Level Densitometer Order PMC71-1D390/0 S/N L904D21509C				
High Level Densitometer Order PMC71-1D390/0 S/N L904D31509C				
Vacuum Gage Order PMC71-1D382/0 S/N L903891509C				
Brine Feed Pump 2SS1G5F5				
Fine Loop Pump Goulds 3657 S/N B1601301				

Circulation Pump EP60-18-326T S/N 0516336025				
Discharge Pump Goulds 3657 S/N B1601302				
Vacuum Pump Flowserve LPHR 45317 AB AAB 0E 0 S/N CA1711194-01				

2. *Acceptance Testing*

Expected	Actual	Pass/Fail	Verified By	Date
The elbow pump is turning in the correct direction	The elbow pump _____ turning in the correct direction			
Fill the crystallizer with fluid to the correct operating level	The crystallizer _____ filled with fluid to the correct operating level			
Flow is coming out in the headspace	Flow _____ coming out in the headspace			
Ensure the brine feed flow meter arrow is pointing towards the circulation loop	The brine feed flow meter arrow _____ pointing towards the circulation loop			
The brine feed pump is turning in the correct direction	The brine feed pump _____ turning in the correct direction			
The brine feed to the crystallizer shows a non-zero value	Brine feed flow:			
The vacuum controller is in manual mode	The vacuum controller _____ in manual mode			
The vacuum controller valve cycles normally	The vacuum controller valve _____ normally			
The vacuum system is on	The vacuum system _____ on			
Both water and steam are present	Both water and steam ____ present			

The pressure drops when the vacuum system is operational	The pressure _____ when the vacuum system is operational			
The discharge pump is turning in the correct direction	The discharge pump _____ turning in the correct direction			
Open the discharge valves	The discharge valves _____ open			
Fluid is being pumped to the centrifuge	Fluid _____ being pumped to the centrifuge			
Put the fine salt loop steam controller in manual mode	The fine salt loop steam controller ____ in manual mode			
The fine salt loop steam valve cycles normally	The fine salt loop steam valve _____ normally			
Enter a fine salt loop temperature of 41 °C	Temperature setpoint:			
The flow through the fine salt loop is non-zero	The flow through the fine sale loop _____ non-zero			
Steam is applied when the temperature drops below the acceptable range	Steam _____ applied when the temperature drops below the acceptable range			
The mother liquor flow meter arrow is pointed to the circulation loop	The mother liquor flow meter arrow _____ pointed to the circulation loop			
Open the supply valve	The supply valve _____ open			
The flow meter reads a non-zero flow	The flow meter _____ a non-zero flow			

3. *Acceptance of Testing and Review*

Expected	Actual	Pass/Fail	Verified By	Date
All actual results match the expected values.	All actual results _____ the expected values.			
The IQ section is complete with no deviations	The IQ section ____ complete with no deviations			
Results reviewed and accepted by				

XII. Performance Qualification (PQ)

1. *Equipment*

Device	Calibration Date	Calibration Expiration	Verified By	Date
Multimeter Model: Fluke 114 S/N: 36250117WS				

Expected	Actual	Pass/Fail	Verified By	Date
Crystallizer #6				
Brine Feed Flow Meter Order 53P40-EL0B1AA0BAAA S/N L80AB116000				
Discharge Flow Meter Order 53P40-EL0B1AA0BAAA S/N A5061716000				
Fines Loop Flow Meter/TT Order 83S25-1V70/0 S/N L9042816000				
Mother Liquor Flow Meter Order 53W25-UL0B1AA0BAAA S/N L904F916000				
Low Level Densitometer Order PMC71-1D390/0 S/N L904D21509C				
High Level Densitometer Order PMC71-1D390/0 S/N L904D31509C				
Vacuum Gage Order PMC71-1D382/0 S/N L903891509C				
Brine Feed Pump 2SS1G5F5				
Fine Loop Pump Goulds 3657 S/N B1601301				

Circulation Pump **EP60-18-326T** **S/N 0516336025**				
Discharge Pump **Goulds 3657** **S/N B1601302**				
Vacuum Pump **Flowserve LPHR 45317 AB AAB 0E 0** **S/N CA1711194-01**				

2. *Acceptance Testing*

Fill and run Crystallizer #6 according to procedure for at least 24 hours prior to making observations. The crystallizer should be in equilibrium during the measurement collection.

a. Vacuum System

Observation Number	Expected	Observation Time	Actual	Pass/Fail	Observed By	Date
1	≤ 1.5 in Hg					
2	≤ 1.5 in Hg					
3	≤ 1.5 in Hg					
4	≤ 1.5 in Hg					
5	≤ 1.5 in Hg					
6	≤ 1.5 in Hg					
7	≤ 1.5 in Hg					
8	≤ 1.5 in Hg					
9	≤ 1.5 in Hg					

b. Fine Salt Reduction System

Observation Number	Expected	Observation Time	Actual	Pass/Fail	Observed By	Date
1	> 15 gpm					
2	> 15 gpm					
3	> 15 gpm					
4	> 15 gpm					
5	> 15 gpm					
6	> 15 gpm					
7	> 15 gpm					
8	> 15 gpm					
9	> 15 gpm					

Observation Number	Expected	Observation Time	Actual	Pass/Fail	Observed By	Date
1	41 ± 3 °C					
2	41 ± 3 °C					
3	41 ± 3 °C					
4	41 ± 3 °C					
5	41 ± 3 °C					
6	41 ± 3 °C					
7	41 ± 3 °C					
8	41 ± 3 °C					
9	41 ± 3 °C					

c. Discharge System

Observation Number	Expected	Observation Time	Actual	Pass/Fail	Observed By	Date
1	22 ± 3 gpm					
2	22 ± 3 gpm					
3	22 ± 3 gpm					
4	22 ± 3 gpm					
5	22 ± 3 gpm					
6	22 ± 3 gpm					
7	22 ± 3 gpm					
8	22 ± 3 gpm					
9	22 ± 3 gpm					

d. Brine Feed System

Observation Number	Expected	Observation Time	Actual	Pass/Fail	Observed By	Date
1	25 ± 3 gpm					
2	25 ± 3 gpm					
3	25 ± 3 gpm					
4	25 ± 3 gpm					
5	25 ± 3 gpm					
6	25 ± 3 gpm					
7	25 ± 3 gpm					
8	25 ± 3 gpm					
9	25 ± 3 gpm					

e. Mother Liquor Feed System

Observation Number	Expected	Observation Time	Actual	Pass/Fail	Observed By	Date
1	> 0 gpm					
2	> 0 gpm					
3	> 0 gpm					
4	> 0 gpm					
5	> 0 gpm					
6	> 0 gpm					
7	> 0 gpm					
8	> 0 gpm					
9	> 0 gpm					

f. USP Testing

Expected	Actual	Pass/Fail	Verified By	Date
USP Testing prior to Crytallizer #6 start collected				
USP Testing after Crystallizer #6 start collected				
Both sets of data pass USP standard testing				

3. *Acceptance of Testing and Review*

Expected	Actual	Pass/Fail	Verified By	Date
All actual results match the expected values.	All actual results _____ the expected values.			
The IQ section is complete with no deviations	The IQ section ____ complete with no deviations			
The OQ section is complete with no deviations	The OQ section ____ complete with no deviations			
Results reviewed and accepted by				

XIII. Protocol Deviation Report Log

Log each protocol deviation report in the table below. Attach PDRs to this protocol.

PDR #	Description	Protocol Section	Date Initiated	Date Resolved

XIV. Signature Identification Log

Identify in the table below any personnel involved in the execution of this protocol.

Name	Affiliation	Signature	Initials	Date