I. Approvals

Signing below indicates agreement that the execution of the Installation, Operational and Performance Qualification Protocol (E17-VAL-PIQ-560) for Crystallizer #6, located at 102 Commerce Street, is complete and the process is validated.

Project Member	Functional Area	Signature	Date
Patrick Owen	Engineering		1/16/17
Kenneth Basehore	Engineering		1/16/17
Sammy Henson	Maintenance		1/16/17
Jason Bumgarner	Production		1-16-17
Matt Haynes	Operations		1-16-17
Deborah Durbin	Quality		1-16-17

A copy of the executed protocol will be attached to this report.

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II. Purpose

The purpose of this protocol is to certify with documented evidence Crystallizer #6 functions as intended throughout its anticipated operating ranges. This protocol sets forth the objectives, methodology, documentation, and test activities needed to complete the Installation Qualification (IQ), Operational Qualification (OQ) and Process Qualification (PQ) for Crystallizer #6, located at 102 Commerce Street at the Main Plant production facility.

III. Summary

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 the older types of Crystallizers were removed from the facility.

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.

The following tests were performed:

- During the IQ, each item in the plumbing, instrumentation and utilities was verified to be in place correctly, and that it's specific function was being performed correctly
- During the OQ, the correct plumbing configuration was verified, and liquid flow was tested to ensure the system was assembled according to plan.
- During the PQ, the following measurements were obtained:
  - The vacuum system remained at < 1.5 in Hg for each measurement
  - The fine salt flow rate remained at > 15 gpm for each measurement
  - The fine salt temperature range remained at 41 ± 3 °C for each measurement
  - The discharge flow rate remained at 22 ± 3 gpm for each measurement
  - The brine feed flow rate remained at 25 ± 3 gpm for each measurement
  - The mother liquor flow rate remained at > 0 gpm for each measurement

Details of the measurements are included in the Summary section of this report.

All USP testing sampled before and after the Crystallizer was installed show passing results for the finished product.

All installation, operational and performance acceptance criteria were met as displayed in the attached executed protocol.

IV. Conclusion

The results of the completed installation, operational and performance qualification protocol show that all acceptance criteria were met for all samples. All testing results provide documented evidence that Crystallizer #6 is installed, is operating and is performing as expected.

V. Recommendations

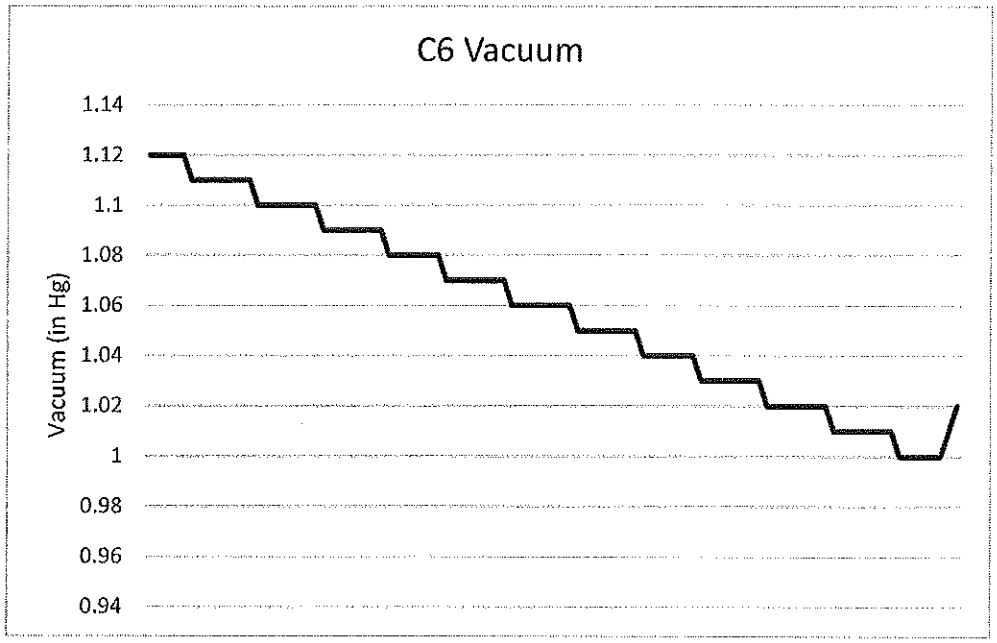
It is recommended that Crystallizer #6, located at the Giles Chemical Main Plant at 102 Commerce Street, Waynesville, NC 28786 be considered validated based on meeting the acceptance criteria of the IQ/OQ/PQ protocol.

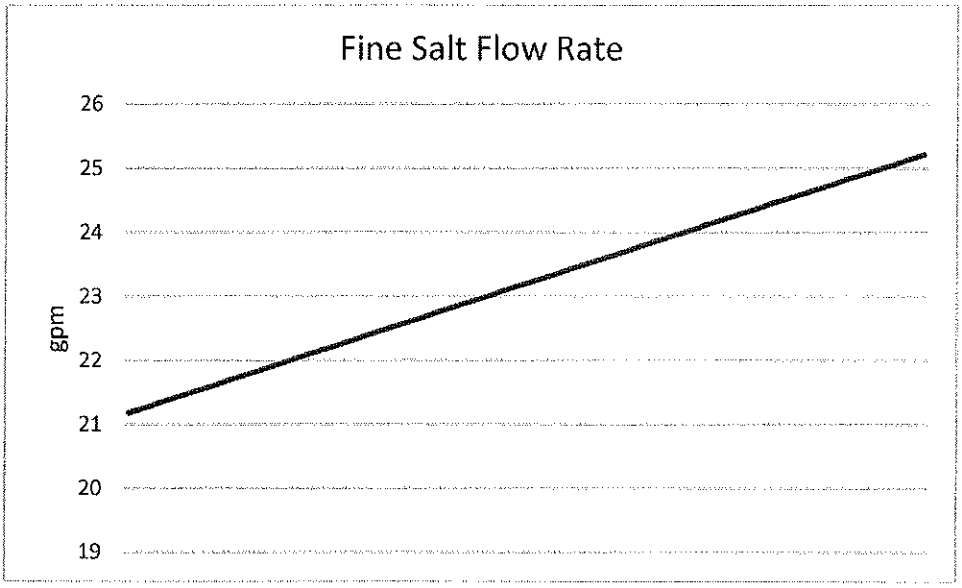
VI. References

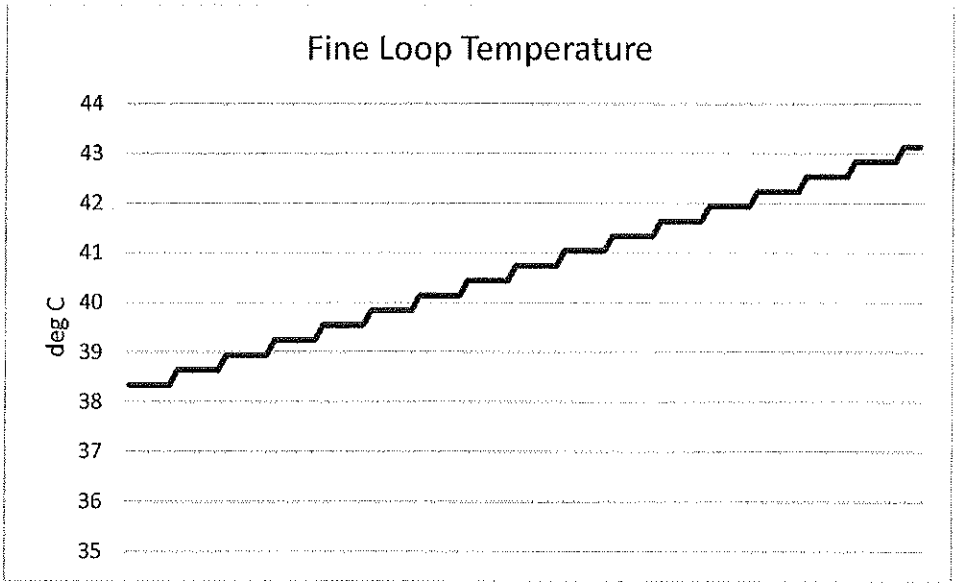
E17-VAL-PIQ-300:	Crystallizer 6 Validation Protocol
P12-PR-200-028:	Filling and Starting a Crystallizer
P12-PR-200-026:	Crystallizer Operation
P12-PR-200-030:	Fine Salt Loop

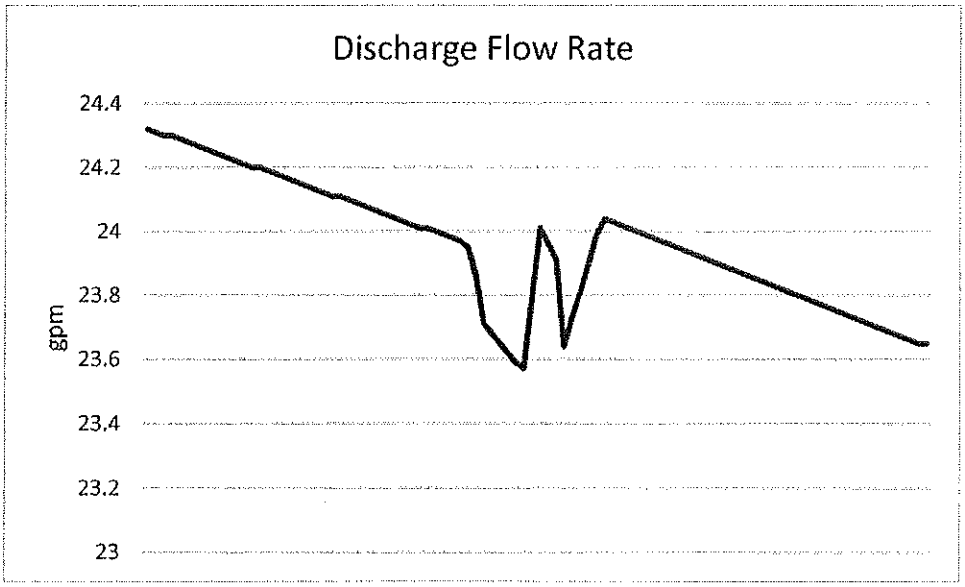
VII. Summary of Process Data collected

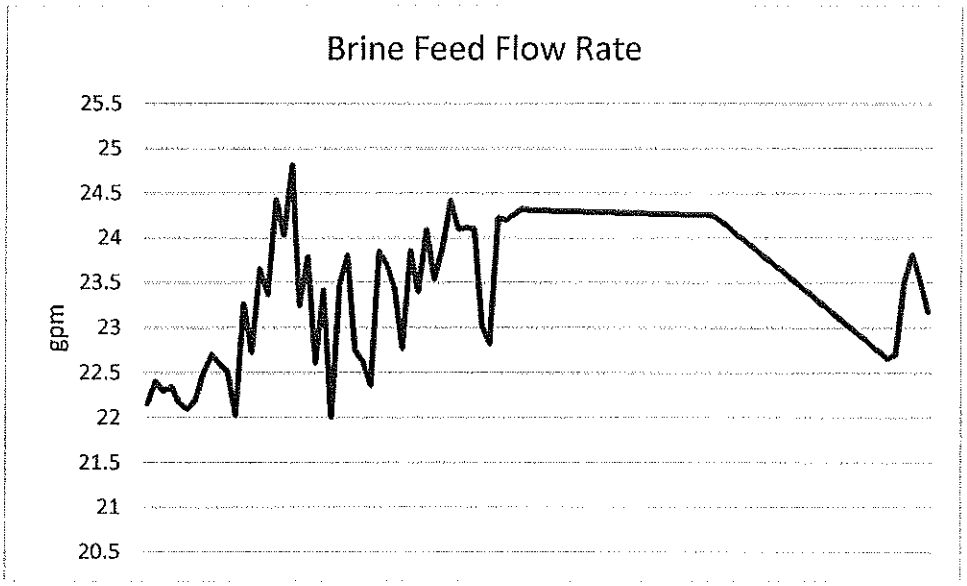
Values were recorded during the PQ portion of the validation every 30 minutes for 4 hours by visually looking at the gages. After the recording was complete, the DCS data was pulled and graphed in order to show data at a much higher than required frequency. The graphs of the data are below:

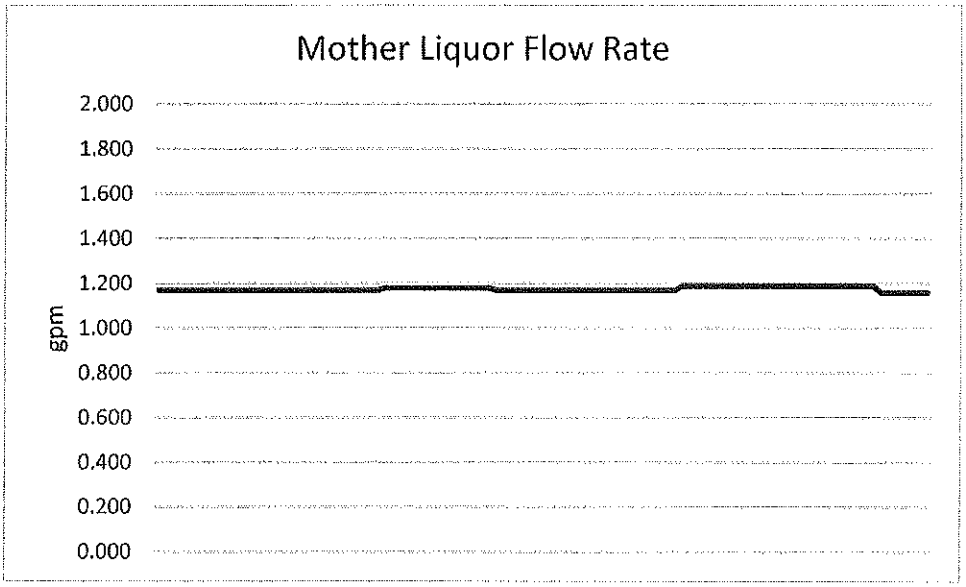












I. Approvals

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	10/2016	10/2017	KLB	1/4/17

Expected	Actual	Pass/Fail	Verified By	Date
Crystallizer #6	CRYSTALLIZER #6	PASS	KLB	1/4/17
Brine Feed Flow Meter Order 53P40-EL0B1AA0BAAA S/N L80AB116000	BRINE FEED FLOW METER ORDER 53P40-EL0B1AA0BAAA S/N L80AB116000	PASS	KLB	1/4/17
Discharge Flow Meter Order 53P40-EL0B1AA0BAAA S/N A5061716000	DISCHARGE FLOW METER ORDER 53P40-EL0B1AA0BAAA S/N A5061716000	PASS	KLB	1/4/17
Fines Loop Flow Meter/TT Order 83S25-1V70/0 S/N L9042816000	FINES LOOP FLOW METER/TT ORDER 83S25-1V70/0 S/N L9042816000	PASS	KLB	1/4/17
Mother Liquor Flow Meter Order 53W25-UL0B1AA0BAAA S/N L904F916000	MOTHER LIQUOR FLOW METER ORDER 53W25-UL0B1AA0BAAA S/N L904F916000	PASS	KLB	1/4/17
Low Level Densitometer Order PMC71-1D390/0 S/N L904D21509C	LOW LEVEL DENSITOMETER ORDER PMC71-1D390/0 S/N L904D21509C	PASS	KLB	1/4/17
High Level Densitometer Order PMC71-1D390/0 S/N L904D31509C	HIGH LEVEL DENSITOMETER ORDER PMC71-1D390/0 S/N L904D31509C	PASS	KLB	1/4/17
Vacuum Gage Order PMC71-1D382/0 S/N L903891509C	VACUUM GAGE ORDER PMC71-1D382/0 S/N L903891509C	PASS	KLB	1/4/17
Brine Feed Pump 2SS1G5F5	BRINE FEED PUMP 2SS1G5F5	PASS	KLB	1/4/17
Fine Loop Pump Goulds 3657 S/N B1601301	FINE LOOP PUMP GOULDS 3657 S/N B1601301	PASS	KLB	1/4/17

Circulation Pump EP60-18-326T S/N 0516336025	CIRCULATION PUMP EP60-18-326T S/N 0516336025	PASS	KLB	1/4/17
Discharge Pump Goulds 3657 S/N B1601302	DISCHARGE PUMP GOULDS 3657 S/N B1601302	PASS	KLB	1/4/17
Vacuum Pump Flowserve LPHR 45317 AB AAB 0E 0 S/N CA1711194-01	VACUUM PUMP FLOWSERVE LPHR 45317 AB AAB 0E 0 S/N CA1711194-01	PASS	KLB	1/4/17

2. *Acceptance Testing*

Expected	Actual	Pass/Fail	Verified By	Date
There is sufficient room around the vessel for maintenance and operations to work	There IS sufficient room around the vessel for maintenance and operations to work	PASS	KLB	1/4/17
The vessel is level	The vessel IS level	PASS	KLB	1/4/17
All hatches and outlets are tightened, plumbed or blanked	All hatches and outlets ARE tightened, plumbed or blanked	PASS	KLB	1/4/17
The elbow pump is in place	The elbow pump IS in place	PASS	KLB	1/4/17
The circulation loop plumbing is in place	The circulation loop plumbing IS in place	PASS	KLB	1/4/17
The brine feed pump is in place	The brine feed pump IS in place	PASS	KLB	1/4/17
The brine feed plumbing connects the brine feed tank to the circulation loop	The brine feed plumbing CONNECTS the brine feed tank to the circulation loop	PASS	KLB	1/4/17
The fine salt loop pump is in place	The fine salt pump IS in place	PASS	KLB	1/4/17
The fine salt loop plumbing connects the vessel to the circulation loop	The fine salt loop plumbing CONNECTS the vessel to the circulation loop	PASS	KLB	1/4/17
The discharge pump is in place	The discharge pump IS in place	PASS	KLB	1/4/17

The discharge plumbing connects the discharge outlet to the centrifuge	The discharge plumbing CONNECTS the discharge outlet to the centrifuge	PASS	KLB	1/4/17
The mother liquor plumbing connects the mother liquor header to the circulation loop	The mother liquor plumbing CONNECTS the mother liquor header to the circulation loop	PASS	KLB	1/4/17
The large condenser plumbing connects the creek tank to the large condenser	The large condenser plumbing CONNECTS the creek tank to the large condenser	PASS	KLB	1/4/17
The large condenser plumbing connects the large condenser to the hot well	The large condenser plumbing CONNECTS the large condenser to the hot well	PASS	KLB	1/4/17
The small condenser plumbing connects the creek tank to the small condenser	The small condenser plumbing CONNECTS the creek tank to the small condenser	PASS	KLB	1/4/17
The small condenser plumbing connects the small condenser to the hot well	The small condenser plumbing CONNECTS the small condenser to the hot well	PASS	KLB	1/4/17
City water is available to the vacuum pump	City water IS available to the vacuum pump	PASS	KLB	1/4/17
Steam is available to the steam ejector	Steam IS available to the steam ejector	PASS	KLB	1/4/17
The vapor pipe connects the top of the crystallizer to the large condenser	The vapor pipe CONNECTS the top of the crystallizer to the large condenser	PASS	KLB	1/4/17
The internal vent pipe connects the lower part of the vessel to the vessel head	The internal vent pipe CONNECTS the lower part of the vessel to the vessel head	PASS	KLB	1/4/17
The discharge flow meter is in the discharge pipe	The discharge flow meter IS in the discharge pipe	PASS	KLB	1/4/17
The mother liquor flow meter is in the mother liquor feed pipe	The mother liquor flow meter IS in the mother liquor feed pipe	PASS	KLB	1/4/17
The brine feed flow meter is in the brine feed flow pipe	The brine feed flow meter IS in the brine feed flow pipe	PASS	KLB	1/4/17
The fine salt loop flow meter is in the fine salt loop piping	The fine salt loop flow meter IS in the fine salt loop piping	PASS	KLB	1/4/17

The density meters are both installed in the vessel	The density meters ARE both installed in the vessel	PASS	KLB	1/4/17
The vacuum meter is installed in the head of the vessel	The vacuum meter IS installed in the head of the vessel	PASS	KLB	1/4/17
The voltage supplied to the pump drive panel is 460V ± 20 V	464V	PASS	KLB	1/4/17
The voltage supplied to the controls is 115V ± 10V	118V	PASS	KLB	1/4/17
The vacuum control valve has air pressure	The vacuum control valve HAS air pressure	PASS	KLB	1/4/17
The fine salt loop control valve has air pressure	The fine salt loop control valve HAS air pressure	PASS	KLB	1/4/17
The steam ejector has steam	The steam ejector HAS steam	PASS	KLB	1/4/17
The fine salt loop has steam and a condensate return	The fine salt loop HAS steam and a condensate return	PASS	KLB	1/4/17

3. *Acceptance of Testing and Review*

Expected	Actual	Pass/Fail	Verified By	Date
All actual results match the expected values.	All actual results MATCH the expected values.	PASS	KLB	1/4/17
Results reviewed and accepted by			[signature]	1/13/17

XI. Operational Qualification (OQ)

1. *Equipment*

Device	Calibration Date	Calibration Expiration	Verified By	Date
Multimeter Model: Fluke 114 S/N: 36250117WS	10/2016	10/2017	KLB	1/5/17

Expected	Actual	Pass/Fail	Verified By	Date
Crystallizer #6	CRYSTALLIZER #6	PASS	KLB	1/5/17
Brine Feed Flow Meter Order 53P40-EL0B1AA0BAAA S/N L80AB116000	BRINE FEED FLOW METER ORDER 53P40-EL0B1AA0BAAA S/N L80AB116000	PASS	KLB	1/5/17
Discharge Flow Meter Order 53P40-EL0B1AA0BAAA S/N A5061716000	DISCHARGE FLOW METER ORDER 53P40-EL0B1AA0BAAA S/N A5061716000	PASS	KLB	1/5/17
Fines Loop Flow Meter/TT Order 83S25-1V70/0 S/N L9042816000	FINES LOOP FLOW METER/TT ORDER 83S25-1V70/0 S/N L9042816000	PASS	KLB	1/5/17
Mother Liquor Flow Meter Order 53W25-UL0B1AA0BAAA S/N L904F916000	MOTHER LIQUOR FLOW METER ORDER 53W25-UL0B1AA0BAAA S/N L904F916000	PASS	KLB	1/5/17
Low Level Densitometer Order PMC71-1D390/0 S/N L904D21509C	LOW LEVEL DENSITOMETER ORDER PMC71-1D390/0 S/N L904D21509C	PASS	KLB	1/5/17
High Level Densitometer Order PMC71-1D390/0 S/N L904D31509C	HIGH LEVEL DENSITOMETER ORDER PMC71-1D390/0 S/N L904D31509C	PASS	KLB	1/5/17
Vacuum Gage Order PMC71-1D382/0 S/N L903891509C	VACUUM GAGE ORDER PMC71-1D382/0 S/N L903891509C	PASS	KLB	1/5/17
Brine Feed Pump 2SS1G5F5	BRINE FEED PUMP 2SS1G5F5	PASS	KLB	1/5/17
Fine Loop Pump Goulds 3657 S/N B1601301	FINE LOOP PUMP GOULDS 3657 S/N B1601301	PASS	KLB	1/5/17

Circulation Pump EP60-18-326T S/N 0516336025	CIRCULATION PUMP EP60-18-326T S/N 0516336025	PASS	KLB	1/5/17
Discharge Pump Goulds 3657 S/N B1601302	DISCHARGE PUMP GOULDS 3657 S/N B1601302	PASS	KLB	1/5/17
Vacuum Pump Flowserve LPHR 45317 AB AAB 0E 0 S/N CA1711194-01	VACUUM PUMP FLOWSERVE LPHR 45317 AB AAB 0E 0 S/N CA1711194-01	PASS	KLB	1/5/17

2. *Acceptance Testing*

Expected	Actual	Pass/Fail	Verified By	Date
The elbow pump is turning in the correct direction	The elbow pump IS turning in the correct direction	PASS	KLB	1/5/17
Fill the crystallizer with fluid to the correct operating level	The crystallizer IS filled with fluid to the correct operating level	PASS	KLB	1/5/17
Flow is coming out in the headspace	Flow IS coming out in the headspace	PASS	KLB	1/5/17
Ensure the brine feed flow meter arrow is pointing towards the circulation loop	The brine feed flow meter arrow IS pointing towards the circulation loop	PASS	KLB	1/5/17
The brine feed pump is turning in the correct direction	The brine feed pump IS turning in the correct direction	PASS	KLB	1/5/17
The brine feed to the crystallizer shows a non-zero value	Brine feed flow: 25 GPM	PASS	KLB	1/5/17
The vacuum controller is in manual mode	The vacuum controller IS in manual mode	PASS	KLB	1/5/17
The vacuum controller valve cycles normally	The vacuum controller valve CYCLES normally	PASS	KLB	1/5/17
The vacuum system is on	The vacuum system IS on	PASS	KLB	1/5/17
Both water and steam are present	Both water and steam ARE present	PASS	KLB	1/5/17

The pressure drops when the vacuum system is operational	The pressure DROPS when the vacuum system is operational	PASS	KLB	1/5/17
The discharge pump is turning in the correct direction	The discharge pump IS turning in the correct direction	PASS	KLB	1/5/17
Open the discharge valves	The discharge valves ARE open	PASS	KLB	1/5/17
Fluid is being pumped to the centrifuge	Fluid IS being pumped to the centrifuge	PASS	KLB	1/5/17
Put the fine salt loop steam controller in manual mode	The fine salt loop steam controller IS in manual mode	PASS	KLB	1/5/17
The fine salt loop steam valve cycles normally	The fine salt loop steam valve CYCLES normally	PASS	KLB	1/5/17
Enter a fine salt loop temperature of 41 °C	Temperature setpoint: 41°C	PASS	KLB	1/5/17
The flow through the fine salt loop is non-zero	The flow through the fine sale loop IS non-zero	PASS	KLB	1/5/17
Steam is applied when the temperature drops below the acceptable range	Steam IS applied when the temperature drops below the acceptable range	PASS	KLB	1/5/17
The mother liquor flow meter arrow is pointed to the circulation loop	The mother liquor flow meter arrow IS pointed to the circulation loop	PASS	KLB	1/5/17
Open the supply valve	The supply valve IS open	PASS	KLB	1/5/17
The flow meter reads a non-zero flow	The flow meter READS a non-zero flow	PASS	KLB	1/5/17

3. *Acceptance of Testing and Review*

Expected	Actual	Pass/Fail	Verified By	Date
All actual results match the expected values.	All actual results MATCH the expected values.	PASS	KLB	1/5/17
The IQ section is complete with no deviations	The IQ section IS complete with no deviations	PASS	KLB	1/5/17
Results reviewed and accepted by			QW	1/13/17

XII. Performance Qualification (PQ)

1. Equipment

Device	Calibration Date	Calibration Expiration	Verified By	Date
Multimeter Model: Fluke 114 S/N: 36250117WS	10/2016	10/2017	KLB	1/5/17

Expected	Actual	Pass/Fail	Verified By	Date
Crystallizer #6	CRYSTALLIZER #6	PASS	KLB	1/5/17
Brine Feed Flow Meter Order 53P40-EL0B1AA0BAAA S/N L80AB116000	BRINE FEED FLOW METER ORDER 53P40-EL0B1AA0BAAA S/N L80AB116000	PASS	KLB	1/5/17
Discharge Flow Meter Order 53P40-EL0B1AA0BAAA S/N A5061716000	DISCHARGE FLOW METER ORDER 53P40-EL0B1AA0BAAA S/N A5061716000	PASS	KLB	1/5/17
Fines Loop Flow Meter/TT Order 83S25-1V70/0 S/N L9042816000	FINES LOOP FLOW METER/TT ORDER 83S25-1V70/0 S/N L9042816000	PASS	KLB	1/5/17
Mother Liquor Flow Meter Order 53W25-UL0B1AA0BAAA S/N L904F916000	MOTHER LIQUOR FLOW METER ORDER 53W25-UL0B1AA0BAAA S/N L904F916000	PASS	KLB	1/5/17
Low Level Densitometer Order PMC71-1D390/0 S/N L904D21509C	LOW LEVEL DENSITOMETER ORDER PMC71-1D390/0 S/N L904D21509C	PASS	KLB	1/5/17
High Level Densitometer Order PMC71-1D390/0 S/N L904D31509C	HIGH LEVEL DENSITOMETER ORDER PMC71-1D390/0 S/N L904D31509C	PASS	KLB	1/5/17
Vacuum Gage Order PMC71-1D382/0 S/N L903891509C	VACUUM GAGE ORDER PMC71-1D382/0 S/N L903891509C	PASS	KLB	1/5/17
Brine Feed Pump 2SS1G5F5	BRINE FEED PUMP 2SS1G5F5	PASS	KLB	1/5/17
Fine Loop Pump Goulds 3657 S/N B1601301	FINE LOOP PUMP GOULDS 3657 S/N B1601301	PASS	KLB	1/5/17

Circulation Pump **EP60-18-326T** **S/N 0516336025**	CIRCULATION PUMP EP60-18-326T S/N 0516336025	PASS	KLB	1/5/17
Discharge Pump **Goulds 3657** **S/N B1601302**	DISCHARGE PUMP GOULDS 3657 S/N B1601302	PASS	KLB	1/5/17
Vacuum Pump **Flowserve LPHR 45317 AB AAB 0E 0** **S/N CA1711194-01**	VACUUM PUMP FLOWSERVE LPHR 45317 AB AAB 0E 0 S/N CA1711194-01	PASS	KLB	1/5/17

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	9:00AM	1.12	PASS	KLB	1/6/17
2	≤ 1.5 in Hg	9:30AM	1.10	PASS	KLB	1/6/17
3	≤ 1.5 in Hg	10:00AM	1.08	PASS	KLB	1/6/17
4	≤ 1.5 in Hg	10:30AM	1.06	PASS	KLB	1/6/17
5	≤ 1.5 in Hg	11:00AM	1.04	PASS	KLB	1/6/17
6	≤ 1.5 in Hg	11:30AM	1.02	PASS	KLB	1/6/17
7	≤ 1.5 in Hg	12:00PM	1.01	PASS	KLB	1/6/17
8	≤ 1.5 in Hg	12:30PM	1.00	PASS	KLB	1/6/17
9	≤ 1.5 in Hg	1:00PM	1.02	PASS	KLB	1/6/17

b. Fine Salt Reduction System

Observation Number	Expected	Observation Time	Actual	Pass/Fail	Observed By	Date
1	> 15 gpm	9:00AM	21.22	PASS	KLB	1/6/17
2	> 15 gpm	9:30AM	21.8	PASS	KLB	1/6/17
3	> 15 gpm	10:00AM	22.29	PASS	KLB	1/6/17
4	> 15 gpm	10:30AM	22.75	PASS	KLB	1/6/17
5	> 15 gpm	11:00AM	23.20	PASS	KLB	1/6/17
6	> 15 gpm	11:30AM	23.65	PASS	KLB	1/6/17
7	> 15 gpm	12:00PM	24.15	PASS	KLB	1/6/17
8	> 15 gpm	12:30PM	24.89	PASS	KLB	1/6/17
9	> 15 gpm	1:00PM	25.22	PASS	KLB	1/6/17

Observation Number	Expected	Observation Time	Actual	Pass/Fail	Observed By	Date
1	41 ± 3 °C	9:00 AM	38.34	PASS	KLB	1/6/17
2	41 ± 3 °C	9:30 AM	38.94	PASS	KLB	1/6/17
3	41 ± 3 °C	10:00 AM	39.84	PASS	KLB	1/6/17
4	41 ± 3 °C	10:30 AM	40.44	PASS	KLB	1/6/17
5	41 ± 3 °C	11:00 AM	41.04	PASS	KLB	1/6/17
6	41 ± 3 °C	11:30 AM	41.64	PASS	KLB	1/6/17
7	41 ± 3 °C	12:00 PM	42.24	PASS	KLB	1/6/17
8	41 ± 3 °C	12:30 PM	42.54	PASS	KLB	1/6/17
9	41 ± 3 °C	1:00 PM	43.14	PASS	KLB	1/6/17

c. Discharge System

Observation Number	Expected	Observation Time	Actual	Pass/Fail	Observed By	Date
1	22 ± 3 gpm	9:00 AM	24.30	PASS	KLB	1/6/17
2	22 ± 3 gpm	9:30 AM	24.21	PASS	KLB	1/6/17
3	22 ± 3 gpm	10:00 AM	24.09	PASS	KLB	1/6/17
4	22 ± 3 gpm	10:30 AM	23.97	PASS	KLB	1/6/17
5	22 ± 3 gpm	11:00 AM	23.96	PASS	KLB	1/6/17
6	22 ± 3 gpm	11:30 AM	23.97	PASS	KLB	1/6/17
7	22 ± 3 gpm	12:00 PM	23.87	PASS	KLB	1/6/17
8	22 ± 3 gpm	12:30 PM	23.77	PASS	KLB	1/6/17
9	22 ± 3 gpm	1:00 PM	23.65	PASS	KLB	1/6/17

d. Brine Feed System

Observation Number	Expected	Observation Time	Actual	Pass/Fail	Observed By	Date
1	25 ± 3 gpm	9:00 AM	22.16	PASS	KLB	1/6/17
2	25 ± 3 gpm	9:30 AM	22.51	PASS	KLB	1/6/17
3	25 ± 3 gpm	10:00 AM	23.25	PASS	KLB	1/6/17
4	25 ± 3 gpm	10:30 AM	22.77	PASS	KLB	1/6/17
5	25 ± 3 gpm	11:00 AM	23.02	PASS	KLB	1/6/17
6	25 ± 3 gpm	11:30 AM	24.28	PASS	KLB	1/6/17
7	25 ± 3 gpm	12:00 PM	23.68	PASS	KLB	1/6/17
8	25 ± 3 gpm	12:30 PM	22.73	PASS	KLB	1/6/17
9	25 ± 3 gpm	1:00 PM	23.18	PASS	KLB	1/6/17

e. Mother Liquor Feed System

Observation Number	Expected	Observation Time	Actual	Pass/Fail	Observed By	Date
1	> 0 gpm	9:00AM	1.17	PASS	KLB	1/6/17
2	> 0 gpm	9:30AM	1.17	PASS	KLB	1/6/17
3	> 0 gpm	10:00AM	1.17	PASS	KLB	1/6/17
4	> 0 gpm	10:30AM	1.18	PASS	KLB	1/6/17
5	> 0 gpm	11:00AM	1.17	PASS	KLB	1/6/17
6	> 0 gpm	11:30AM	1.17	PASS	KLB	1/6/17
7	> 0 gpm	12:00PM	1.19	PASS	KLB	1/6/17
8	> 0 gpm	12:30PM	1.19	PASS	KLB	1/6/17
9	> 0 gpm	1:00PM	1.16	PASS	KLB	1/6/17

f. USP Testing

Expected	Actual	Pass/Fail	Verified By	Date
USP Testing prior to Crytallizer #6 start collected	DATA COLLECTED 12/19/16 - 12/21/16	PASS	KLB	1/13/17
USP Testing after Crystallizer #6 start collected	DATA COLLECTED 1/9/17 - 1/13/17	PASS	KLB	1/13/17
Both sets of data pass USP standard testing	BOTH SETS OF DATA PASS USP STANDARD TESTING	PASS	KLB	1/13/17

3. Acceptance of Testing and Review

Expected	Actual	Pass/Fail	Verified By	Date
All actual results match the expected values.	All actual results MATCH the expected values.	PASS	KLB	1/13/17
The IQ section is complete with no deviations	The IQ section IS complete with no deviations	PASS	KLB	1/13/17
The OQ section is complete with no deviations	The OQ section IS complete with no deviations	PASS	KLB	1/13/17
Results reviewed and accepted by			QW	1/13/17

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
		N/A	KLB 1/13/17	

XIV. Signature Identification Log

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

Name	Affiliation	Signature	Initials	Date
KENNETH BASEHORE	ENGINEERING	Kenneth D Basehore	KLB	1/4/17
Ashley Williams	Quality	Ashley Williams	AW	1/13/17