

**GILES CHEMICAL ~ PREMIER MAGNESIA****Validation Protocol**

Title: Crystallizer #4 IQ/OQ/PQ Protocol

Number: E13-VAL-PIQ-301

Owner: Patrick Owen



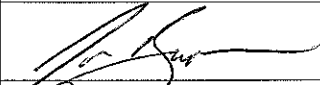
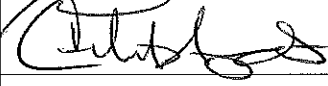

Revision: 0

Effective Date: May 24, 2013

Page: 1 of 23

**Approvals**

Signing below indicates agreement that the protocol is ready for execution of the Installation Protocol for Crystallizer #4 located at 102 Commerce Street, Waynesville, NC at the Manufacturing facility.

Project Team Member	Functional Area	Signature	Date
Patrick Owen	Engineering		5/24/13
Robert Willis	Maintenance		5/24/13
Jason Bumgarner	Production		5-24-13
Matt Haynes	Operations		5-24-13
Deborah Durbin	Quality		5-24-13

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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Title: Crystallizer #4 IQ/OQ/PQ Protocol

Number: E13-VAL-PIQ-301

Owner: Patrick Owen

Revision: 0

Effective Date: May 24, 2013

Page: 2 of 23



TABLE OF CONTENTS		Page #
APPROVAL PAGE		1
TABLE OF CONTENTS		2
I.	PURPOSE	3
II.	BACKGROUND	3
III.	SCOPE	3
IV.	SYSTEM DESCRIPTION	3-4
V.	ROLES AND RESPONSIBILITIES	4-5
VI.	TEST PROGRAM	5-9
A	INSTALLATION QUALIFICATION	5
B	OPERATIONAL QUALIFICATION	7
C	PERFORMANCE QUALIFICATION	8-9
VII.	CALIBRATION	9
VIII.	REFERENCE MATERIAL	9
ATTACHMENT I:	INSTALLATION QUALIFICATION	10
ATTACHMENT II:	OPERATIONAL QUALIFICATION	13
ATTACHMENT III:	PERFORMANCE QUALIFICATION	16
ATTACHMENT IV	CALIBRATION DATA SHEET	20
ATTACHMENT V:	PROTOCOL DEVIATION REPORT LOG	21
ATTACHMENT VI:	PROTOCOL DEVIATION REPORT	22
ATTACHMENT VII	SIGNATURE IDENTIFICATION LOG SHEET	23

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Title: Crystallizer #4 IQ/OQ/PQ Protocol

Number: E13-VAL-PIQ-301

Owner: Patrick Owen

Revision: 0

Effective Date: May 24, 2013

Page: 3 of 23

**I. PURPOSE:**

The purpose of this protocol is to provide documented evidence of the proper installation of Crystallizer #4. This will 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 #4 located in the Manufacturing Building at 102 Commerce Street in Waynesville, NC.

II. BACKGROUND:**2.1 Historical**

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

2.2 Current Project

A Giles Authorization for Expenditure (AFE) was signed on December 27, 2012 to purchase and install Crystallizer #4 by May 1, 2013. The vessel design was an exact copy of #2, and #3 Crystallizers. The machine will Crystallize USP Epsom Salt from Brine produced at Giles' Manufacturing facility.

The products that are impacted by this study are all Epsom Salt products manufactured by Giles Chemical.

III. SCOPE

This study will be performed on Crystallizer #4. 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 current Good Manufacturing Practice (cGMP) requirements.

IV. SYSTEM DESCRIPTION:**1. OVERVIEW**

Crystallizer #4 uses vacuum to cool a continuous stream of saturated brine to form crystals, which are then discharged to a centrifuge.

2. DESCRIPTION OF OPERATION

There are 6 systems in that make up Crystallizer #4. All are in operation when Crystallizer #4 is in operation:

- a. 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, and circulation pump piping.

Controlled Document



GILES CHEMICAL ~ PREMIER MAGNESIA

Validation Protocol

Title: Crystallizer #4 IQ/OQ/PQ Protocol

Number: E13-VAL-PIQ-301

Owner: Patrick Owen

Revision: 0

Effective Date: May 24, 2013

Page: 4 of 23



- b. 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.
- c. 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.
- d. Fine Salt Reduction Loop: The fine salt 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. This 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.
- e. 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.
- f. 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.

V. ROLES AND RESPONSIBILITIES

1. Engineering

- ❖ Write and issue the protocol
- ❖ Investigate protocol deviation reports
- ❖ Execute the OQ and manage the data collection for the PQ.
- ❖ Review raw data and originate interim notification to Quality Assurance
- ❖ Write and route the final report



2. Quality Assurance

- ❖ Review and approve the protocol.
- ❖ Review and approve raw data and notifications.
- ❖ Review, approve, and store the final report.

3. Maintenance

- ❖ Provide Equipment Manuals needed to execute operational qualification.
- ❖ Review and approve the protocol.
- ❖ Execute the IQ.
- ❖ Review and approve raw data and notifications.

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	GILES CHEMICAL ~ PREMIER MAGNESIA		
	Validation Protocol		
	Title: Crystallizer #4 IQ/OQ/PQ Protocol	Number: E13-VAL-PIQ-301	
	Owner: Patrick Owen	Revision: 0	
	Effective Date: May 24, 2013	Page: 5 of 23	

❖ Review and approve the final report

4. Production

❖ Execute the PQ.

❖ Review and approve the final report.

VI. TEST PROGRAM

A. INSTALLATION QUALIFICATION

Objective

The objective of the installation verification is to document that each of the 6 systems that comprise Crystallizer #4 are installed properly and document the components of each system for future reference.

Equipment/Materials

Crystallizer #4

Ideal Digital Multimeter Model #61-340 (SN 100100221)

Level

Procedure

Perform each listed below for Crystallizer #4:

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 instrument is level.
3. Vessel: Ensure that all hatches and outlets are tightened, plumbed, or blanked
4. Plumbing:
 - a. Ensure the Elbow Pump plumbing is in place
 - b. Ensure the Brine Feed Pump and plumbing feeds from the Brine Feed Tank to the Elbow Pump Loop
 - c. Ensure the Fine Salt Loop plumbing feeds from the vessel and returns to the Elbow Pump Loop
 - 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

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GILES CHEMICAL ~ PREMIER MAGNESIA

Validation Protocol

Title: Crystallizer #4 IQ/OQ/PQ Protocol

Number: E13-VAL-PIQ-301

Owner: Patrick Owen

Revision: 0

Effective Date: May 24, 2013

Page: 6 of 23



- 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 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 Line
- 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 plumbing
- e. Ensure the Level and Density, and Vacuum transmitters are installed on the body of the Crystallizer.

6. Utilities

- a. Electrical Requirements:
 - Ensure Voltage is correct to Pump Drive Panel
 - Ensure Voltage is correct to Instrumentation
- b. Air
 - Ensure Vacuum Control Valve has air pressure
 - Ensure Fine Salt Loop Control Valve has air Pressure
- c. Steam
 - Ensure Steam Ejector has steam
 - Ensure Fine Salt Loop has steam and condensate return

Acceptance Criteria

If each item of Plumbing, Instrumentation, and Utilities is in place and allows for the specific function as outlined, then Crystallizer #4 will be considered to be installed properly.

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GILES CHEMICAL ~ PREMIER MAGNESIA

Validation Protocol

Title: Crystallizer #4 IQ/OQ/PQ Protocol

Number: E13-VAL-PIQ-301

Owner: Patrick Owen

Revision: 0

Effective Date: May 24, 2013

Page: 7 of 23



B. OPERATION QUALIFICATION

Objective

The objective of the operational verification is to document that the components of the 6 systems that comprise Crystallizer #4 are operable and oriented correctly for the machine to operate.

Equipment/Materials

Crystallizer #4

Procedure

Perform each listed task for Crystallizer #4 (all motors are to be observed as standing facing the "grill" end of the motor – not the output shaft):

1. Vessel and Circulation
 - a. Ensure Elbow Pump is turning clockwise
 - b. With fluid in the Crystallizer, turn on the circulator and ensure a flow is coming out in the headspace by looking through the top sight glass
2. Feed System
 - a. Ensure flow meter arrow is pointing toward the elbow loop plumbing
 - b. Turn on the brine pump and ensure the motor is turning clockwise
 - c. Ensure brine will feed to Crystallizer and readout shows a flow
3. Vacuum System
 - a. Put controller in manual and verify that the vacuum valve will open and close
 - b. With fluid in the Crystallizer, turn on the vacuum system and ensure that it has both water and steam
 - c. Check the vacuum sensor to determine that pressure drops when the system is on
4. Discharge System
 - a. Ensure the pump motor is turning clockwise when the pump is on.
 - b. With the vessel full open the valves and turn on the discharge pump. With the system set point on 22 gpm observe if flow comes out at the centrifuge
5. Fine Salt Reduction System
 - a. Put controller in manual and verify that steam valve will open and close
 - b. With the vessel full, turn on the fine salt loop pump and enter a set point of 41 C in the controller. Verify with the flow meter that there is flow and that the temperature rises when steam is called for.
6. Mother Liquor System

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Validation Protocol

Title: Crystallizer #4 IQ/OQ/PQ Protocol

Number: E13-VAL-PIQ-301

Owner: Patrick Owen

Revision: 0

Effective Date: May 24, 2013

Page: 8 of 23



- a. Ensure the flow meter arrow is pointing toward the direction of the flow (from ML tank to Crystallizer)
- b. Open valves and ensure Mother Liquor flows to the Crystallizer and verify that the flow meter reads a flow.

Acceptance Criteria

Verification that each motor is rotating in the correct direction, each flowmeter is oriented so as to give a positive flow reading, and control valves can open and close when the unit is in operation.

C. PERFORMANCE QUALIFICATION

Objective

The objective of performance testing is to document that Crystallizer #4 performs the functions required by Giles Chemical.

- The vacuum pulled by the vacuum system is sufficient to cool the brine solution to 35 degrees C
- That the Fines Reduction Loop will heat a flow of at least 15 gallons per minute to 41 +/- 3 degrees C
- That the discharge system will discharge 22 +/- 3 gallons per minute for at least 1 hour without stopping.
- That the plant product USP Testing results will pass with Crystallizer #5 running.

Equipment/Materials

Crystallizer #4

Brine

Mother Liquor

Calibrated Instrumentation Installed on the Crystallizer

Procedure

Fill and run #4 Crystallizer according to procedure for at least 24 hours before making observations.

Perform each listed task for Crystallizer #4:

1. Vacuum
 - a. Observe vacuum reading every 30 minutes for 4 hours
2. Fines Reduction Loop
 - a. Observe Fines Reduction Loop flow and temperature every 30 minutes for 4 hours

Controlled Document



GILES CHEMICAL ~ PREMIER MAGNESIA

Validation Protocol

Title: Crystallizer #4 IQ/OQ/PQ Protocol

Number: E13-VAL-PIQ-301

Owner: Patrick Owen

Revision: 0

Effective Date: May 24, 2013

Page: 9 of 23



3. Discharge System

- a. Observe the Discharge flow every 30 minutes for 4 hours

4. Plant USP Testing

- a. Obtain a product sample and test for USP parameters in the QA Laboratory

Acceptance Criteria

Vacuum absolute pressure cannot exceed 1.50 inches of Mercury in any observation

Fines Reduction Loop flow cannot be less than 15.0 gallons per minute in any observation

Fines Reduction Loop temperature cannot be outside of the range of 38.0 – 44.0 degrees Celsius in any observation

Discharge flow cannot be outside the range of 19-25 gallons per minute in any observation

USP testing of final product must pass in all parameters

VII. CALIBRATION

Verify that all instrumentation is calibrated at the time of installation.

- Ideal Digital Multimeter Model #61-340 (SN 100100221)
- Discharge Flow Meter
- Brine Feed Flow Meter
- Level Probe
- Vacuum Probe
- Fines Reduction Flow and Temperature Meter

VIII. REFERENCE:

P12-PR-200-026 rev 3 "Crystallizer Operations" Giles Chemical, 2012

P12-PR-200-028 rev 6 "Filling and Starting a Crystallizer" Giles Chemical, 2012

Controlled Document

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Title: Crystallizer #4 IQ/OQ/PQ Protocol

Number: E13-VAL-PIQ-301

Owner: Patrick Owen

Revision: 0

Effective Date: May 24, 2013

Page: 10 of 23

**CRYSTALLIZER #4: INSTALLATION QUALIFICATION****A. Installation Qualification****1. Location**

LOCATION			
Distance Criterion	Is the current area sufficient to allow access without obstructions (Yes/No)	Verified By	Date
Allow sufficient room around the machine for Maintenance and Operations to perform their duties			
Comments:			

2. Level

Verify that the instrument is level

LEVEL			
Is the unit level? (Yes/No)	Acceptable (Yes/No)	Verified By	Date
Comments:			

3. Vessel

VESSEL			
Criterion	Are all hatches and outlets tightened, plumbed, or blanked (Yes/No)	Verified By	Date
Ensure all hatches and outlets are tightened, plumbed, or blanked			
Comments:			

Reviewed By: _____

Date: _____

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Title: Crystallizer #4 IQ/OQ/PQ Protocol

Number: E13-VAL-PIQ-301

Owner: Patrick Owen

Revision: 0

Effective Date: May 24, 2013

Page: 11 of 23

**4. Plumbing**

Plumbing			
Specified Location	Plumbing present and connected to proper inlet and outlet (Yes/No)	Verified By	Date
Elbow Pump plumbing			
Brine Feed Line			
Fine Salt Loop Lines			
Discharge Line			
Mother Liquor Line			
Large Condenser Lines			
Small Condenser Lines			
Vacuum Pump plumbing			
Ejector Steam Line			
Vapor Pipe			
Vent Pipe			
Comments:			

Reviewed By: _____

Date: _____

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Title: Crystallizer #4 IQ/OQ/PQ Protocol

Number: E13-VAL-PIQ-301

Owner: Patrick Owen

Revision: 0

Effective Date: May 24, 2013

Page: 12 of 23

**5. Instrumentation****Instrumentation**

Specified Location	Instrument present and oriented properly (Yes/No)	Instrument Serial Number	Verified By	Date
Discharge Flow Meter				
Mother Liquor Flow Meter				
Brine Feed Flow Meter				
Fine Salt Loop Flow Meter				
Density Transmitter				
Level Transmitter				
Vacuum Transmitter				
Comments:				

6. Utilities

- a. Verify that unit is receiving its specified utility requirements.

UTILITIES**Electrical**

Specified	Actual	Verified By	Date
460 V +/- 20V for Pump Drive Panel			
115V +/- 10 for Controls			
Air			
Air Present for Vacuum Control Valve			
Air Present for Fine Salt Loop Control Valve			
Steam			
Steam Supply for Ejector			
Fine Salt Loop Steam			
Comments:			

Reviewed By: _____**Date:** _____**Controlled Document**

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Title: Crystallizer #4 IQ/OQ/PQ Protocol

Number: E13-VAL-PIQ-301

Owner: Patrick Owen

Revision: 0

Effective Date: May 24, 2013

Page: 13 of 23

**CRYSTALLIZER #4: OPERATIONAL QUALIFICATION****B. Operational Qualification****1. Vessel and Circulation**

Vessel and Circulation				
Description	Function	Did Item function properly (Yes/No)	Verified By	Date
Elbow Pump	Elbow Pump turns clockwise as viewed from the motor grill when the drive is "on"			
Circulation	With vessel filled with brine and Elbow Pump on circulation discharge should be visible from the top sight glass			
Comments:				

2. Feed System

Feed System				
Description	Function	Did Item function properly (Yes/No)	Verified By	Date
Brine Flow Meter	Flow meter directional arrow should point toward Elbow Loop plumbing			
Brine Pump	With Brine Pump on the motor should turn clockwise as viewed from the motor grill when the drive is "on"			
Brine Flow Meter	With valves open and pump on the Brine Flow Meter on the Brine Flow Meter should register a flow			
Comments:				

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Date: _____

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Title: Crystallizer #4 IQ/OQ/PQ Protocol

Number: E13-VAL-PIQ-301

Owner: Patrick Owen

Revision: 0

Effective Date: May 24, 2013

Page: 14 of 23

**3. Vacuum System**

Vacuum System				
Description	Function	Did Item function properly (Yes/No)	Verified By	Date
Vacuum Control Valve	With controller in Manual, valve should close at 0% and be fully open at 100%			
Vacuum System Utilities	Ensure steam is on Ejector and Water is on both Condensers			
Vacuum Sensor	When Vacuum System is on, the Vacuum Sensor should show the pressure dropping			
Comments:				

4. Discharge System

Discharge System				
Description	Function	Did Item function properly (Yes/No)	Verified By	Date
Discharge Pump Motor	Ensure pump motor turns clockwise as viewed from the motor grill when the drive is "on"			
Discharge Flow	With vessel full, open both valves and put system set point on 22 gpm. Verify flow at Centrifuge.			
Comments:				

Reviewed By: _____

Date: _____

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Title: Crystallizer #4 IQ/OQ/PQ Protocol

Number: E13-VAL-PIQ-301

Owner: Patrick Owen

Revision: 0

Effective Date: May 24, 2013

Page: 15 of 23

**5. Fine Salt Reduction System**

Fine Salt Reduction System				
Description	Function	Did Item function properly (Yes/No)	Verified By	Date
Steam Control Valve	With controller in Manual, valve should close at 0% and be fully open at 100%			
Fine Salt Flow	Ensure flow meter shows flow with FS Loop pump on			
Steam Control	Ensure temperature in Fine Salt Loop rises when steam is called for			
Comments:				

6. Mother Liquor System

Mother Liquor System				
Description	Function	Did Item function properly (Yes/No)	Verified By	Date
Flow Meter	Ensure Flow Meter arrow points away from the Mother Liquor Tank and toward the Crystallizer			
Flow	Ensure that when valves are open that flow is observed on the Flow Meter			
Comments:				

Reviewed By: _____

Date: _____

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Title: Crystallizer #4 IQ/OQ/PQ Protocol

Number: E13-VAL-PIQ-301

Owner: Patrick Owen

Revision: 0

Effective Date: May 24, 2013

Page: 16 of 23

**CRYSTALLIZER #4: PERFORMANCE QUALIFICATION**

C. Performance Qualification – to be performed with Crystallizer #4 operating for at least 24 hours and at steady state

1. Vacuum

Performance Qualification Data				
Time	Vacuum (< or = 1.50 inches Hg)	Did Item Meet Criteria (Yes/No)	Verified By	Date
Comments:				

Reviewed By: _____

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Number: E13-VAL-PIQ-301

Owner: Patrick Owen

Revision: 0

Effective Date: May 24, 2013

Page: 17 of 23

**2. Fines Reduction Loop Flow**

Performance Qualification Data				
Time	Fines Reduction Loop Flow (Not Less than 15.0 gallons per minute)	Did Item Meet Criteria (Yes/No)	Verified By	Date
Comments:				

Reviewed By: _____

Date: _____

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Number: E13-VAL-PIQ-301

Owner: Patrick Owen

Revision: 0

Effective Date: May 24, 2013

Page: 18 of 23

**3. Fines Reduction Loop Temperature**

Performance Qualification Data				
Time	Fines Reduction Loop Temperature (38.0 – 44.0 Degrees C)	Did Item Meet Criteria (Yes/No)	Verified By	Date
Comments:				

Reviewed By: _____

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Number: E13-VAL-PIQ-301

Owner: Patrick Owen

Revision: 0

Effective Date: May 24, 2013

Page: 19 of 23

**4. Discharge Flow**

Performance Qualification Data				
Time	Discharge Flow (19 – 25 gallons per minute)	Did Item Meet Criteria (Yes/No)	Verified By	Date
Comments:				

5. USP Testing

Date and Time Sample Taken : _____ Verified By: _____

Results: PASS / FAIL (circle one) Verified By: _____ Date: _____

Note: Attach USP Testing Results to the Final Report

Reviewed By: _____ Date: _____

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Number: E13-VAL-PIQ-301

Owner: Patrick Owen

Revision: 0

Effective Date: May 24, 2013

Page: 20 of 23

**VII. CALIBRATION VERIFICATION**

Equipment	Manufacturer	Model Number	Serial Number	Calibration Date	Verified By	Date
Multimeter						
Discharge Flow Meter						
Brine Feed Flow Meter						
Level Probe						
Vacuum Probe						
Fines Reduction Flow Meter						

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ATTACHMENT I - PROTOCOL DEVIATION REPORT LOG

Log each Protocol Deviation Report in the table below. Attach the PDRs to this Attachment.

[illegible]

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	Validation Protocol		
	Title: Crystallizer #4 IQ/OQ/PQ Protocol	Number: E13-VAL-PIQ-301	
	Owner: Patrick Owen	Revision: 0	
	Effective Date: May 24, 2013	Page: 22 of 23	

ATTACHMENT II. PROTOCOL DEVIATION REPORT (PDR)

_____ General Information _____

System Name: _____ Protocol Number: _____

Deviation Report Number: _____ Protocol Step & Page No.: _____

_____ Instructions _____

1. The validation specialist assigns a sequential report number for each deviation with a specific protocol. For example, 001, 002, etc. can be easily referenced in a report.
2. Reference the relevant protocol number, step and page number of the noted deviation above.
3. Complete the below listed sections. If necessary, use additional pages and attach any supporting info.
4. Include the original PDR(s) with the protocol as an attachment. Summarize the impact of the deviation in the Validation Report.

Description of Deviation:



Investigation Evaluation and Results:

Corrective Action and Resolution:

Overall Investigation Review:

Prepared By: _____ Date: _____

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	Validation Protocol		
	Title: Crystallizer #4 IQ/OQ/PQ Protocol	Number: E13-VAL-PIQ-301	
	Owner: Patrick Owen	Revision: 0	
	Effective Date: May 24, 2013	Page: 23 of 23	

ATTACHMENT III - SIGNATURE IDENTIFICATION LOG SHEET

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

Name	Affiliation	Signature	Initial	Date

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