Approvals

Signing below indicates agreement that the protocol is ready for execution of the Installation Qualification 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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1.0 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) for Crystallizer #4 located in the Manufacturing Building at 102 Commerce Street in Waynesville, NC..

2.0 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 Crystallizers were removed from the facility.

2.2 Current Project

A Giles Authorization for Expenditure (AFE) was signed on December 27, 2012 to install Crystallizer #4 by May 1, 2013. The vessel design was an exact copy of #2 and #3 Crystallizers (#1 was a used Crystallizer optimized for Sodium Sulfate). 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.

3.0 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. The executed protocol will verify that all acceptance criteria have been met, and that the Crystallizer meets current Good Manufacturing Practice (cGMP) requirements.

4.0 SYSTEM DESCRIPTION:

4.1 OVERVIEW

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

4.2 DESCRIPTION OF OPERATION

Brine Feed: A continuous stream of brine is pumped into the Circulation Loop of the Crystallizer. The Circulation Loop is pulled from the top of the sealed vessel and contains mother liquor and very small crystals.

Circulation: The Crystallizer cools a continuous flow of saturated brine to supersaturation while using a high flow of small crystals and mother liquor to fluidize a bed of larger crystals. The mother liquor and small crystals are pulled from the top of the fluidized bed and recirculated internally. The saturated brine is introduced into the fluidizing stream.

Evaporation: Cooling is achieved by evaporation. The evaporation is from a constant vacuum being pulled on the entire system. A 3-stage system of barometric condenser, steam ejector, and vacuum pump is used to pull vacuum.

Fines Destruction: Very fine crystals from the top of the fluidized bed are removed, pumped through a heat exchanger for melting, and returned to the circulation loop. This increases the average size of the crystals discharged.

Discharge: A stream of crystal slurry is constantly being pulled out near the bottom so the largest crystals are removed.

5.0 ROLES AND RESPONSIBILITIES

5.1 Engineering/Maintenance

- Write and issue the protocol
- Investigate protocol deviation reports
- Provide documentation as necessary
- Execute the protocol

5.2 Quality Assurance

- Review and approve the protocol.
- Review and approve any exceptions and resolutions
- Store the approved protocol and the executed protocol.

5.3 Production

- Assist with execution of the IQ
- Review and approve the protocol

6.0 GENERAL PROCEDURE

This Installation Qualification of Crystallizer #4 at GILES CHEMICAL, Waynesville, NC will be executed by completing the forms, tables and test sheets provided in this protocol. These sheets will serve to document that Crystallizer #4 is compliant with design and use expectations.

Completion of the Installation Qualification will be governed by the following general procedures:

- In the execution of the Installation Qualification for existing systems, field conditions will be verified against specifications. These specifications will be taken from purchase orders, design and functional specifications, and lists of materials where those documents are available. In the event that no supporting documentation or existing specification is available for a characteristic that is deemed necessary, the specification will be listed, N/AV (None Available), and the existing condition will be documented as baseline information.
- Verification methods should first be documents that have been field verified or information obtained from nameplate data. When this is not possible, using vendor supplied user manuals is sufficient.

- The forms and tables provided in the protocol are to be completely filled out to provide as much detail as possible – no blank spaces.
- All protocols will be completed using black/blue indelible ink.
- Opaque liquefied (whiteout) correction fluid cannot be used on this document.
- Make any corrections to hand-written data by striking out with a single line and initialing and dating the correction.
- With the exception of the named approvers, each person who performs or reviews any section of the testing within this document should complete all information required on the Signature Identification Sheet.
- For all tests that require the executor to make a comparison, calculation or a judgment of satisfactory completion, the test case will include a "Yes/Exception #" column. This section will require the executor to record the disposition of each test or test step as appropriate.
- Discrepancies may be observed as the protocol is being executed. Should this occur, a "Protocol Deviation Report" is to be prepared. The "Protocol Deviation Report" identifies the discrepancy found, determines the impact, and corrective action. It also identifies the exception as "critical" or "non-critical." A non-critical exception will have no impact to the system or testing. In conjunction with the Quality Unit, a plan of action will be determined and an appropriate corrective action will be taken. Critical exceptions must be closed out before execution of the corresponding section of the protocol may continue. All exceptions must be closed prior to the final approval of the executed protocol and final summary report.
- All test instruments utilized in the execution of this protocol must include calibration certification and be traceable to NIST standards.
- Any comments regarding the test are to be recorded on the data sheets under the "Comments" section. If there are no comments or exceptions, the section will be crossed out with a single line, initialed and dated.
- The individual completing the form will sign and date the "Completed by" line at the bottom. If more than one individual provided entries, each individual will initial and date his/her entry, and sign and date the bottom.
- A reviewer, who has read and agrees with the execution and conclusions, will sign the "Reviewed by" signature line.
- If additional form pages are required, copies of the form pages can be inserted into the protocol and given the appropriate page numbers.

7.0 GENERAL ACCEPTANCE CRITERIA

The acceptance criteria will be considered met if all execution steps in that test section pass. If a test case does not meet expectations, the test may be repeated after proper Protocol Deviation Report documentation and corrective action. The re-test data will be included as an attachment to the referenced Protocol Deviation Report.

For closure of the Installation Qualification, all sections will be successfully completed and all exceptions will have been addressed and resolved. Once these steps have been accomplished, the Installation Qualification will be considered completed and closed.

8.0 CALIBRATION

Verify that all instrumentation used for validation that requires calibration is calibrated.

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

CRYSTALLIZER #4: INSTALLATION QUALIFICATION

9.0 Major Component List

9.1 PURPOSE

The purpose of this section is to verify that all major components associated with the Crystallizer are included and the "as-found" conditions are recorded.

13.2 PROCEDURE

Verify that each component is installed and in the proper location.

Complete the following tables that define each of the major components of the Crystallizer. Document the following where applicable:

Component Name
Manufacturer
Model Number
Serial Number
Location

Materials of Construction

13.3 ACCEPTANCE CRITERIA

- Each component is located and identified
- Wetted surface materials of construction are suitable for use with Magnesium Sulfate (plastic, stainless steel, or brass)

9.4 Component List

COMPONENT	MANUFACTURER	MODEL	SERIAL#	LOCATION (FLOOR)	MATERIAL OF CONSTRUCTION	VERIFY BY	DATE
VESSEL							
BRINE FEED PUMP							
FINE SALT LOOP PUMP							
DISCHARGE PUMP							
BAROMETRIC CONDENSER							
STEAM EJECTOR							
VACUUM PUMP							
CIRCULATION PUMP							
COMMENTS							

Acceptance Criteria met? (Yes/Exception #) _____

Reviewed By: _____ **Date:** _____

10.0 Location and Utilities

10.1 Location

a. Verify that Crystallizer #4 is placed properly for operation.

LOCATION			
Distance Criterion	**Is the current area sufficient to open the access without obstructions (Yes/No)**	**Verified By**	**Date**
Allow sufficient room around the machine for access doors and panels to be opened			
The machine must be located in an area that is adequately ventilated			
Comments:			

10.2 Level

a. Make sure that the Crystallizer is level.

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

10.3 Utilities

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

Utilities			
Electrical			
Specified	**Actual**	**Verified By**	**Date**
460 V for Circulator Pump			
115V for Controls			
24V for Controls			
Water and Steam			
The machine requires cooling water and steam			
Is cooling water available via pump?			
Is steam system connected?			
Comments:			

Reviewed By: _____ **Date:** _____

11.0 Instrumentation

11.1 PURPOSE

The purpose of this section is to verify that instrumentation associated with the Crystallizer is installed and calibrated as applicable at installation.

11.2 PROCEDURE

Verify that each instrument is installed and in the proper location.

Complete the following tables that define each of the instruments of the Crystallizer. Document the following where applicable:

Component Name
Manufacturer
Model Number
Serial Number
Location

Calibration

11.3 ACCEPTANCE CRITERIA

- Each component is located and identified
- As applicable, calibration certificates are available for each instrument (not applicable for some devices ie. RTD and thermocouples).

11.4 Instrument List

COMPONENT	MANUFACTURER	MODEL	SERIAL#	LOCATION (FLOOR)	CALIBRATION CERTIFICATE AVAILABLE	VERIFY BY	DATE
BRINE FLOW METER							
ML FLOW METER							
VACUUM METER							
LEVEL METER							
DENSITY METER							
FINE SALT FLOW METER/ TEMPERATURE							
CRYSTALLIZER TEMPERATURE PROBE							
CIRCULATION PUMP							
DISCHARGE FLOW METER							
COMMENTS							

Acceptance Criteria met? (Yes/Exception #) _____

Reviewed By: _____ **Date:** _____

12.0 SOP Verification

12.1 PURPOSE

The purpose of this section is to verify that standard operating procedures (SOP's) associated with the Crystallizer are available in at least DRAFT form.

12.2 PROCEDURE

Verify that each SOP exists.

Complete the following tables that define each of the SOP's for the Crystallizer. Document the following where applicable:

Document Title
Document Number
Version
Date

12.3 ACCEPTANCE CRITERIA

- Each SOP is at least in Draft form
- All applicable procedures are listed for documentation verification.

12.4 DOCUMENT LIST

TITLE	DOCUMENT NUMBER	VERSION	DATE	LOCATION	VERIFY BY	DATE
COMMENTS						

Acceptance Criteria met? (Yes/Exception #) _____

Reviewed By: _____ **Date:** _____

13.0 CALIBRATION VERIFICATION

Equipment	Serial #	Calibration Date	Calibration Due Date	Verified By	Date
Multimeter					

Reviewed By: _____ **Date:** _____

ATTACHMENT I - PROTOCOL DEVIATION REPORT LOG

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

PDR #	DESCRIPTION	DATE INITIATED	DATE RESOLVED
Comments:			

ATTACHMENT III - PROTOCOL DEVIATION REPORT (PDR)

General Information

System Name: _____ Protocol Number: _____

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

Instructions

1. A sequential report number for each deviation with a specific protocol is assigned. 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: _____

Quality Approved By: _____ Date: _____

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