**Approvals**

Signing below indicates agreement that the execution of the Installation, Operational, and Performance Qualification Protocol for the Epsom Salt Dryer/Cooler system located at 102 Commerce Street at the Manufacturing facility is complete and the process is validated.

| Project Team Member | Functional Area | Signature | Date |
|---|---|---|---|
| Patrick Owen | Engineering | | 3/2/16 |
| Sammy Henson | Maintenance | | 3/2/16 |
| Jason Bumgarner | Production | | 3-2-16 |
| Matt Haynes | Operations | | 3-2-16 |
| Deborah Durbin | Quality | | 3-2-16 |

The executed protocol will be attached behind the report.

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## I. PURPOSE

The purpose of this report is to provide documented evidence that Epsom Salt Dryer/Cooler is installed properly and functions as intended throughout its anticipated operating range. This final report provides documented evidence that the objectives, methodology, documentation, and test activities needed to complete the Installation Qualification (IQ), Operational Qualification (OQ), and Performance Qualification (PQ) for the Dryer/Cooler located in the Manufacturing Building at 102 Commerce Street in Waynesville, NC were all executed and all acceptance criteria were met.

## II. SUMMARY

The first Carman Industries Dryer Section was installed in June of 2011 and the second Dryer Section was installed in July of 2011. The Cooler Section was installed in January of 2016.

The products that are impacted by this study are all Epsom Salt products manufactured by Giles Chemical. No other departments or systems were affected by the installation or use of this equipment.

The following tests were performed in this qualification:

Physical Installation Verification – level, voltage, presence of utilities

Controls Verification – blower controls, control valve range, shaker controls

Performance Verification – will the system produce a product with the correct Loss-on-Ignition and at a final salt temperature of less than 30 degrees C

All Installation, Operational, and Performance acceptance criteria were met as displayed in the tables in the Appendices.

## III. CONCLUSION

The results of the executed protocol verify that all acceptance criteria have been met. All testing results provide documentation that the Dryer/Cooler is installed, operating, and performing as expected. The Dryer/Cooler system is considered to be validated.

## IV. RECOMMENDATIONS

It is recommended that the Dryer/Cooler located in the Manufacturing Building at 102 Commerce Street in Waynesville, NC be considered validated based on meeting all acceptance criteria of the IQ/OQ/PQ Protocol.

## V. REFERENCE

*E16-VAL-PIQ-421, Dryer/Cooler IQ/OQ/PQ Protocol, rev 0, 1/13/2016 Giles Chemical*

# APPENDIX I: DRYER/COOLER INSTALLATION QUALIFICATION

## A. Installation Qualification

### 01. Location

| LOCATION | |
|---|---|
| **Distance Criterion** | **Is the current area sufficient to allow access without obstructions (Yes/No)** |
| Allow sufficient room around the machine for Maintenance and Operations to perform their duties | YES |

### 02. Level

**Verify that the machine is level**

| LEVEL | |
|---|---|
| **Unit** | **Is the unit level? (Yes/No)** |
| Dryer Zone 1 | YES |
| Dryer Zone 2 | YES |
| Cooler | YES |

### 03. Electrical and Plumbing

| Electrical | |
|---|---|
| **Specified Location** | **Connected Properly? (Yes/No)** |
| Dryer Zone 1 Blower | YES |
| Dryer Zone 2 Blower | YES |
| Cooler Blower | YES |
| Exhaust Fan 1 | YES |
| Exhaust Fan 2 | YES |
| Dryer Zone 1 Shaker | YES |
| Dryer Zone 2 Shaker | YES |
| Cooler Shaker | YES |
| **Plumbing** | |
| Dryer Zone 1 Steam | YES |
| Dryer Zone 1 Condensate | YES |
| Dryer Zone 2 Steam | YES |
| Dryer Zone 2 Condensate | YES |
| Cooler Chill Water | YES |
| **Physical Connections** | |
| Dryer Boot in place? | YES |
| Cooler Boot in place? | YES |
| Sample Port on Dryer? | YES |
| Sample Port on Cooler? | YES |

### 04. Instrumentation

| Instrumentation | |
|---|---|
| **Specified Location** | **Instrument present and oriented properly (Yes/No)** |
| Dryer Zone 1 Airbox RTD | YES |
| Dryer Zone 2 Airbox RTD | YES |
| Cooler Zone RTD | YES |
| Dryer Salt RTD | YES |
| Cooler Salt RTD | YES |
| Dryer Zone 1 Controller Readout | YES |
| Dryer Zone 2 Controller Readout | YES |
| Cooler Controller Readout | YES |

### 05. Utilities

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

| UTILITIES | |
|---|---|
| **Electrical** | |
| **Specified** | **Actual** |
| 460 V +/- 20V Drive Panel 1 | 468 V |
| 460 V +/- 20V Drive Panel 2 | 468 V |
| 115V +/- 10 for Controls | 119 V |
| **Air** | |
| Air Present for Control Valves? | YES |
| **Steam** | |
| Steam Supply Drying Present? | YES |
| Condensate Return Present? | YES |
| Cooling Water Present? | YES |

# APPENDIX II: DRYER/COOLER OPERATIONAL QUALIFICATION

## B. Operational Qualification

### 01. Steam Coil

| Steam Coil and Controls | | |
|---|---|---|
| **Description** | **Function** | **Did Item function properly (Yes/No)** |
| Steam 0% | Is valve at 0% with controller set on 0%? | YES |
| Steam 100% | Is valve at 100% with controller set on 100%? | YES |

### 02. Cooling Coil

| Cooling Coil and Controls | | |
|---|---|---|
| **Description** | **Function** | **Did Item function properly (Yes/No)** |
| Cooling 0% | Is valve at 0% with controller set on 0%? | YES |
| Cooling 100% | Is valve at 100% with controller set on 100%? | YES |

### 03. Shakers

| Shakers | | |
|---|---|---|
| **Description** | **Function** | **Did Item function properly (Yes/No)** |
| Dryer Zone 1 | When actuated, does bed shake smoothly? | YES |
| Dryer Zone 2 | When actuated, does bed shake smoothly? | YES |
| Cooler | When actuated, does bed shake smoothly? | YES |

**04. Blowers**

| Blowers | | |
|---|---|---|
| **Description** | **Function** | **Did Item function properly (Yes/No)** |
| Dryer Zone 1 | Does blower run when switched on? | YES |
| Dryer Zone 2 | Does blower run when switched on? | YES |
| Cooler | Does blower run when switched on? | YES |
| Exhaust 1 | Does blower run when switched on? | YES |
| Exhaust 2 | Does blower run when switched on? | YES |

# APPENDIX III: DRYER/COOLER PERFORMANCE QUALIFICATION

**A. Performance Qualification – to be performed with Dryer/Cooler operating for at least 30 minutes and at steady state**

| Performance Qualification Data | | |
|---|---|---|
| **Time** | **Cooler Salt Exit Temperature (< 30 Degrees C)** | **Did Item Meet Criteria (Yes/No)** |
| 09:30 AM | 24.6 C | YES |
| 10:02 AM | 25.0 C | YES |
| 10:33 AM | 24.9 C | YES |
| 11:08 AM | 25.4 C | YES |
| 11:39 AM | 25.5 C | YES |
| 12:05 PM | 26.0 C | YES |

**B. USP Testing**

**LOI (LOSS ON INGITION) RESULT: ____50.52%_____**
**LOI (LOSS ON IGNITION) SPECIFICATION RANGE: _____40.0% - 52.0%_____**
**Results: PASS**

**Approvals**

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

| Project Team Member | Functional Area | Signature | Date |
|---|---|---|---|
| Patrick Owen | Engineering | | 1/13/16 |
| Sammy Henson | Maintenance | | 1/13/16 |
| Jason Bumgarner | Production | | 1-13-16 |
| Matt Haynes | Operations | | 1-13-16 |
| Deborah Durbin | Quality | | 1-13-16 |

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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## 1.0 PURPOSE:

The purpose of this protocol is to provide documented evidence of the proper installation of the Dryer/Cooler. 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 the Dryer/Cooler 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. During most of that time, Rotary Kiln type dryers were used to dry the salt. The only drying specification for USP is for Loss on Ignition (LOI). Giles has always met the specification for LOI with the rotary dryers. To reduce caking (which is not a specification) Giles chose to install a fluidized bed Dryer and Cooler to replace the Rotary Dryers.

### 2.2 Current Project

The first Carman Industries Dryer Section was installed in June of 2011 and the second Dryer Section was installed in July of 2011. The Cooler Section was installed in January of 2016.

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 the Dryer/Cooler System. 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 Dryer/Cooler meets current Good Manufacturing Practice (cGMP) requirements.

## 4.0 SYSTEM DESCRIPTION:

### 4.1 OVERVIEW

The Dryer/Cooler system uses air heated with steam coils (indirect steam heat) to fluidize and dry the Epsom Salt Crystals produced by the Crystallizers. The Cooler Section uses air cooled with chilled water coils (indirect cooling) to cool the salt before it enters the packaging equipment.

### 4.2 DESCRIPTION OF OPERATION

Moist salt crystals are introduced into the Dryer/Cooler Section by a screw conveyor carrying the moist salt from the Centrifuges. Heated air is blown up through a perforated plate in the Dryer Sections. The heated air partially suspends the salt crystals and removes surface moisture from them. After the salt is dried, cool air is blown up through a perforated plate in the Cooler Section. The chilled air cools the salt before discharging the salt a screw conveyor.

There are 4 basic systems in operation when the Dryer/Cooler is in operation:

1. Steam coils use a supply of steam generated by our boilers to heat the filtered ambient air for drying the Crystals.
2. The chilled water coil cools the filtered ambient air in order to cool the crystals after they have passed from the Dryer sections into the Cooler section
3. The blowers supply air to and evacuate air from the Dryer and Cooler sections for drying, cooling and fluidizing the salt crystals.
4. The shakers apply small amplitude directional vibration to the sections to help convey the fluidized salt crystals.

## 5.0 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.
   - ❖ Review and approve the final report
4. Production
   - ❖ Execute the PQ.
   - ❖ Review and approve the final report.

## 6.0 TEST PROGRAM

### A. INSTALLATION QUALIFICATION

Objective

The objective of the installation verification is to document that each of the systems that comprise Dryer/Cooler are installed properly and document the components of each system for future reference.

Equipment/Materials

Dryer/Cooler System

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

Level

Procedure

Perform each listed below for the Dryer/Cooler:

- Location: Verify that the equipment is situated to allow sufficient room around the machine for Maintenance and Operations to perform their respective duties.
- Level: Verify Sections are level.
- Electrical and Plumbing:
  - Ensure electrical disconnects are mounted for each motor
  - Ensure Steam and Condensate plumbing is attached the steam and condensate headers.
  - Ensure inlets and outlets are connected with flexible boots.
  - Ensure that there is a sample port in each outlet.
- Instrumentation
  - Ensure that the manual air pressure gauges are connected to taps above and below the beds.
  - Ensure the air temperature RTD's are in the air boxes.
  - Ensure the salt temperature RTD's are placed so as to measure the temperature of the salt flow.
  - Ensure the temperature readouts display the temperatures.
- Utilities
  - Electrical Requirements:
    - Ensure Voltage is correct to the Dryer Panel.
  - Steam and Cooling Water
    - Steam and condensate are plumbed to heated sections.
    - Ensure cooling water is plumbed to cooler section.

Acceptance Criteria

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

## B. OPERATION QUALIFICATION

Objective

The objective of the operational verification is to document that the components of the systems that comprise the Dryer/Cooler are operable for the machine to operate.

Equipment/Materials

Dryer/Cooler

Procedure

Perform each listed task for the Dryer/Cooler System:

1. Steam Coil and Controls
    a. Place the heated zone controller in manual and set to 0%
    b. Verify the steam valve position at 0%
    c. Place the heated zone controller in manual and set to 100%
    d. Verify the steam valve position at 100%
2. Cooling Coil and Controls
    a. Place the cooling zone controller in manual and set to 0%
    b. Verify the cooling water valve position at 0%
    c. Place the cooling zone controller in manual and set to 100%
    d. Verify the cooling water valve position at 100%
3. Shakers
    a. Turn on Dryer Zone #1 shaker
    b. Verify the Dryer Zone #1 bed shakes smoothly.
    c. Turn on Dryer Zone #2 shaker
    d. Verify the Dryer Zone #2 bed shakes smoothly.
    e. Turn on the Cooler Zone shaker
    f. Verify the Cooler Zone bed shakes smoothly
    g. Turn off all shakers

4. Blowers
    a. Turn on Dryer Zone #1 blower.
    b. Turn on Dryer Zone #2 blower.
    c. Turn on Exhaust Fan #1.
    d. Verify the blowers are running.
    e. Turn on the Cooler Zone blower.
    f. Turn on Exhaust Fan #2.
    g. Verify the blowers are running.
    h. Turn off all blowers.

Acceptance Criteria

Verification that each control works, the shakers operate, and the blowers operate indicate that the system is operational.

## C. PERFORMANCE QUALIFICATION

Objective

The objective of performance testing is to document that Dryer/Cooler performs the functions required by Giles Chemical.

- The salt is dried to USP Loss-on-Ignition specification.
- The salt is cooled to below 30 degrees C at the exit of the Cooler.

Equipment/Materials

Dryer/Cooler

Production Salt

Thermolyne Pyrometer (Serial # 76KA0319) with k-thermocouple

Lab test results

Procedure

Start up and run the Dryer/Cooler. Start production salt flow through the Dryer/Cooler and run for at least 30 minutes.

1. Measure the salt temperature at the exit port of the Dryer/Cooler in 30 minute intervals for 2 hours.
2. Plant USP Testing
    a. Obtain a product sample and test for USP parameters in the QA Laboratory

Acceptance Criteria

Dryer/Cooler exit salt temperature cannot be higher than 30.0 degrees Celsius in any observation.

USP testing of final product must pass in all parameters

## 7.0 CALIBRATION

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

- Ideal Digital Multimeter Model #61-340 (SN 100100221)
- Thermolyne Pyrometer Model PM20700 (SN 76KA0319)

## 8.0 REFERENCE:

P12-PR-200-085 rev 2 "Salt Cooler Start Up and Shut Down" Giles Chemical, 2016

# DRYER/COOLER : INSTALLATION QUALIFICATION

## A. Installation Qualification

### 01. 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 | Yes | Per | 2/24/16 |
| **Comments:** | | | |

Per 2/24/16

### 02. Level

**Verify that the machine is level**

| LEVEL | | | |
|---|---|---|---|
| **Unit** | **Is the unit level? (Yes/No)** | **Verified By** | **Date** |
| Dryer Zone 1 | Yes | Per | 2/24/16 |
| Dryer Zone 2 | Yes | Per | 2/24/16 |
| Cooler | Yes | Per | 2/24/16 |
| **Comments:** | | | |

Per 2/24/16

**Reviewed By:** _____ **Date:** 2/29/16

## 03. Electrical and Plumbing

| Electrical | | | |
|---|---|---|---|
| **Specified Location** | **Connected Properly? (Yes/No)** | **Verified By** | **Date** |
| Dryer Zone 1 Blower | Yes | PG | 2/24/16 |
| Dryer Zone 2 Blower | Yes | PG | 2/24/16 |
| Cooler Blower | Yes | PG | 2/24/16 |
| Exhaust Fan 1 | Yes | PG | 2/24/16 |
| Exhaust Fan 2 | Yes | PG | 2/24/16 |
| Dryer Zone 1 Shaker | Yes | PG | 2/24/16 |
| Dryer Zone 2 Shaker | Yes | PG | 2/24/16 |
| Cooler Shaker | Yes | PG | 2/24/16 |
| **Plumbing** | | | |
| Dryer Zone 1 Steam | Yes | PG | 2/24/16 |
| Dryer Zone 1 Condensate | Yes | PG | 2/24/16 |
| Dryer Zone 2 Steam | Yes | PG | 2/24/16 |
| Dryer Zone 2 Condensate | Yes | PG | 2/24/16 |
| Cooler Chill Water | Yes | PG | 2/24/16 |
| **Physical Connections** | | | |
| Dryer Boot in place? | Yes | PG | 2/24/16 |
| Cooler Boot in place? | Yes | PG | 2/24/16 |
| Sample Port on Dryer? | Yes | PG | 2/24/16 |
| Sample Port on Cooler? | Yes | PG | 2/24/16 |
| **Comments:** | | | |

PG 2/24/16

**Reviewed By:** _____ **Date:** 2/29/16

## 04. Instrumentation

| Instrumentation | | | |
|---|---|---|---|
| **Specified Location** | **Instrument present and oriented properly (Yes/No)** | **Verified By** | **Date** |
| Dryer Zone 1 Airbox RTD | Yes | PGV | 2/24/16 |
| Dryer Zone 2 Airbox RTD | Yes | PGV | 2/24/16 |
| Cooler Zone RTD | Yes | PGV | 2/24/16 |
| Dryer Salt RTD | Yes | PGV | 2/24/16 |
| Cooler Salt RTD | Yes | PGV | 2/24/16 |
| Dryer Zone 1 Controller Readout | Yes | PGV | 2/24/16 |
| Dryer Zone 2 Controller Readout | Yes | PGV | 2/24/16 |
| Cooler Controller Readout | Yes | PGV | 2/24/16 |
| **Comments:** | | | |

PGV 2/24/16

**Reviewed By:** _____ **Date:** 2/29/16

**05. Utilities**

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

| UTILITIES | | | |
|---|---|---|---|
| **Electrical** | | | |
| **Specified** | **Actual** | **Verified By** | **Date** |
| 460 V +/- 20V Drive Panel 1 | 468V | PC | 2/24/16 |
| 460 V +/- 20V Drive Panel 2 | 468V | PC | 2/24/16 |
| 115V +/- 10 for Controls | 119V | PC | 2/24/16 |
| **Air** | | | |
| Air Present for Control Valves? | Yes | PC | 2/24/16 |
| **Steam** | | | |
| Steam Supply Drying Present? | Yes | PC | 2/24/16 |
| Condensate Return Present? | Yes | PC | 2/24/16 |
| Cooling Water Present? | Yes | PC | 2/24/16 |
| **Comments:** | | | |

PC 2/24/16

**Reviewed By:** _____ **Date:** 2/29/16

# DRYER/COOLER: OPERATIONAL QUALIFICATION

## B. Operational Qualification

### 01. Steam Coil

| Steam Coil and Controls | | | | |
|---|---|---|---|---|
| **Description** | **Function** | **Did Item function properly (Yes/No)** | **Verified By** | **Date** |
| Steam 0% | Is valve at 0% with controller set on 0%? | Yes | PC | 2/24/16 |
| Steam 100% | Is valve at 100% with controller set on 100%? | Yes | PC | 2/24/16 |
| **Comments:** | | | | |

PC 2/24/16

### 02. Cooling Coil

| Cooling Coil and Controls | | | | |
|---|---|---|---|---|
| **Description** | **Function** | **Did Item function properly (Yes/No)** | **Verified By** | **Date** |
| Cooling 0% | Is valve at 0% with controller set on 0%? | Yes | PC | 2/24/16 |
| Cooling 100% | Is valve at 100% with controller set on 100%? | Yes | PC | 2/24/16 |
| **Comments:** | | | | |

PC 2/24/16

**Reviewed By:** _____ **Date:** 2/25/16

### 03. Shakers

| Shakers | | | | |
|---|---|---|---|---|
| **Description** | **Function** | **Did Item function properly (Yes/No)** | **Verified By** | **Date** |
| Dryer Zone 1 | When actuated, does bed shake smoothly? | Yes | PS | 2/24/16 |
| Dryer Zone 2 | When actuated, does bed shake smoothly? | Yes | PS | 2/24/16 |
| Cooler | When actuated, does bed shake smoothly? | Yes | PS | 2/24/16 |
| **Comments:** | | | | |

per 2/24/16

### 04. Blowers

| Blowers | | | | |
|---|---|---|---|---|
| **Description** | **Function** | **Did Item function properly (Yes/No)** | **Verified By** | **Date** |
| Dryer Zone 1 | Does blower run when switched on? | Yes | PS | 2/24/16 |
| Dryer Zone 2 | Does blower run when switched on? | Yes | PS | 2/24/16 |
| Cooler | Does blower run when switched on? | Yes | PS | 2/24/16 |
| Exhaust 1 | Does blower run when switched on? | Yes | PS | 2/24/16 |
| Exhaust 2 | Does blower run when switched on? | Yes | PS | 2/24/16 |
| **Comments:** | | | | |

PS 2/24/16

**Reviewed By:** _____ **Date:** 2/28/16

# DRYER/COOLER: PERFORMANCE QUALIFICATION

**C. Performance Qualification – to be performed with Dryer/Cooler operating for at least 30 minutes and at steady state**

**01. Vacuum**

| Performance Qualification Data | | | | |
|---|---|---|---|---|
| **Time** | **Cooler Salt Exit Temperature (< 30 Degrees C)** | **Did Item Meet Criteria (Yes/No)** | **Verified By** | **Date** |
| 0930 | 24.6 °C | Yes | PW | 2/25/16 |
| 1002 | 25.0 °C | Yes | PW | 2/25/16 |
| 1033 | 24.9 °C | Yes | PW | 2/25/16 |
| 1108 | 25.4 °C | Yes | PW | 2/25/16 |
| 1139 | 25.5 °C | Yes | PW | 2/25/16 |
| 1205 | 26.0°C | Yes | PW | 2/25/16 |
| **Comments:** | | | | |

PW 2/25/16

**Reviewed By:** _____ **Date:** 2/29/16

**02. USP Testing**

**Date and Time Sample Taken :** 2/25/2016 0810am **Verified By:** PO

**Results:** (PASS) **FAIL (circle one)** **Verified By:** PO **Date:** 2/27/2016

**Note: Attach USP Testing Results to the Final Report**

**Reviewed By:** [signature] **Date:** 2/29/16

## VII. CALIBRATION VERIFICATION

| Equipment | Manufacturer | Model Number | Serial Number | Calibration Date | Verified By | Date |
|---|---|---|---|---|---|---|
| Multimeter | Ideal | 61-340 | 100100221 | at factory | Per | 2/24/16 |
| Pyrometer | Thermolyne | PM20700 | 76MA0319 | at factory | Per | 2/24/16 |

**Reviewed By:** _____ **Date:** 2/29/16

**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 |
|---|---|---|---|
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| **Comments:** | | | |

PDR 2/25/16

## ATTACHMENT II. PROTOCOL DEVIATION REPORT (PDR)

per 2/25/16

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: _____

**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 |
|---|---|---|---|---|
| Patrick Owen | Engr. : Maint. Mgr. | | PO | 2/24/16 |
| Brook Vaughn | Quality | | BV | 2/29/16 |
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From Page No. BER ① Entry error 2/25/14 JJ

| LOI | crucible # | crucible mass | mass + Sample | mass after Dry | LOI |
|---|---|---|---|---|---|
| Sample 1 2/25/14 07-04/14 dryer | P | 14.9540 | 15.9628 | 15.4532 | 50.52 % |
| Sample 2 2/24/14 160055 BER A2 | My | 14.5973 | 15.5229 | 15.0255 | ~~51.04~~ 50.93 % |
| Sample 3 2/24/14 160055 BER A4 | 27 | 16.1108 | 17.1850 | 16.6364 | 51.07 % |
| Sample 4 2/24/14 160055 BER M1 | 23 | 16.9652 | 17.9100 | 17.4271 | 51.05 % |
| Sample 5 2/24/14 160055 BER M2 | DW | 15.7543 | 16.7282 | 16.2314 | 51.01 % |
| Sample 6 2/24/14 160055 BER C1 | 25 | 17.5317 | 18.5310 | 18.0200 | 51.14 % |
| Sample 7 2/24/14 160055 BER C2 | 22 | 18.6338 | 19.6921 | 19.1522 | 51.02 % |
| Sample 8 2/24/14 160055 END A2 | JH | 16.5612 | 17.5258 | 17.0343 | 50.90 % |
| Sample 9 2/24/14 160055 END A4 | # | 15.4539 | 16.4427 | 16.1382 | 51.02 % |
| Sample 10 2/24/14 160055 END M1 | YY | 15.0530 | 16.0014 | 15.5178 | 51.00 % |
| Sample 11 2/24/14 160055 END M2 | D | 16.6704 | 17.6545 | 17.1537 | 50.99 % |
| Sample 12 2/24/14 160055 END C1 | QH | 15.1457 | 16.1499 | 15.6383 | 50.95 % |
| Sample 13 2/24/14 160055 END C2 | ✓ | 16.0710 | 17.0217 | 16.5391 | 51.09 % |

| Identification | Tube 1 | Tube 2 | Tube 3 | Assay | Initial | Final | % mg/dL |
|---|---|---|---|---|---|---|---|
| Sample 1 | | | | Sample 1 | 0.00mL | 41.52mL | 99.95 % |
| Sample 2 | | | | Sample 2 | 0.00mL | 41.28mL | 99.37 % |
| Sample 3 | | | | Sample 3 | 0.00mL | 41.22mL | 99.22 % |
| Sample 4 | | | | Sample 4 | 0.00mL | 41.38mL | 99.61 % |
| Sample 5 | | | | Sample 5 | 0.00mL | 41.36mL | 99.56 % |
| Sample 6 | | | | Sample 6 | 0.00mL | 41.34mL | 99.51 % |
| Sample 7 | | | | Sample 7 | 0.00mL | 41.33mL | 99.49 % |
| Sample 8 | | | | Sample 8 | 0.00mL | 41.30mL | 99.42 % |
| Sample 9 | | | | Sample 9 | 0.00mL | 41.34mL | 99.53 % |
| Sample 10 | | | | Sample 10 | 0.00mL | 41.27mL | 99.35 % |
| Sample 11 | | | | Sample 11 | 0.00mL | 41.25mL | 99.30 % |
| Sample 12 | | | | Sample 12 | 0.00mL | 41.22mL | 99.22 % |
| Sample 13 | | | | Sample 13 | 0.00mL | 41.28mL | 99.37 % |

pH

| | | |
|---|---|---|
| Sample 1 = 7.00 | Sample 6 = 8.46 | Sample 11 = 8.37 |
| Sample 2 = 7.06 | Sample 7 = 7.37 | Sample 12 = 8.29 |
| Sample 3 = 8.22 | Sample 8 = 7.57 | Sample 13 = 7.80 |
| Sample 4 = 8.51 | Sample 9 = 7.51 | |
| Sample 5 = 8.21 | Sample 10 = 8.08 | |

JJ 2/25/14

To Page No.

| Witnessed & Understood by me, | Date | Invented by: | Date |
|---|---|---|---|
| | 2/27/14 | Recorded by: | 11/25/14 |

JS 2/25/16

## USP Testing 02/25/16

Method: USP1 Operator: Admin Date Printed: 25 Feb 2016 11:44:16

| ITEM | | | | |
|---|---|---|---|---|
| QC1 - 1 | | | | |
| 25 Feb 2016 10:34:59 | | | | |
| Line | As 193.759 | Cd 226.502 | Hg 194.227 | Pb 220.353 |
| Conc. | 0.02421 | 0.49594 | 0.29917 | 0.09979 |
| Line | Fe 259.940 | Ni 231.604 | Se 196.090 | Cl 134.724 x |
| Conc. | 7.93373 | 9.92497 | 12.05599 | 48.11285 |
| 2/25/16 #1 - 1 | | | | |
| 25 Feb 2016 10:37:03 | | | | |
| Line | As 193.759 | Cd 226.502 | Hg 194.227 | Pb 220.353 |
| Conc. | 0.05730 | -0.01475 | 0.02851 | 0.06409 |
| Line | Fe 259.940 | Ni 231.604 | Se 196.090 | Cl 134.724 x |
| Conc. | -0.00869 | -0.22102 | 0.71214 | 69.56822 |
| 2/25/16 #2 - 1 | | | | |
| 25 Feb 2016 10:39:06 | | | | |
| Line | As 193.759 | Cd 226.502 | Hg 194.227 | Pb 220.353 |
| Conc. | 0.03605 | -0.01934 | 0.02947 | 0.01400 |
| Line | Fe 259.940 | Ni 231.604 | Se 196.090 | Cl 134.724 x |
| Conc. | -0.02514 | -0.18978 | 0.14844 | 51.28171 |
| 2/25/16 #3 - 1 | | | | |
| 25 Feb 2016 10:41:09 | | | | |
| Line | As 193.759 | Cd 226.502 | Hg 194.227 | Pb 220.353 |
| Conc. | 0.05232 | -0.01914 | 0.04123 | 0.06604 |
| Line | Fe 259.940 | Ni 231.604 | Se 196.090 | Cl 134.724 x |
| Conc. | -0.00773 | -0.27251 | 0.04712 | 73.03689 |
| 2/25/16 #4 - 1 | | | | |
| 25 Feb 2016 10:43:13 | | | | |
| Line | As 193.759 | Cd 226.502 | Hg 194.227 | Pb 220.353 |
| Conc. | 0.06096 | -0.01500 | 0.03251 | 0.07630 |
| Line | Fe 259.940 | Ni 231.604 | Se 196.090 | Cl 134.724 x |
| Conc. | -0.03334 | -0.26106 | 0.06375 | 61.45417 |
| 2/25/16 #5 - 1 | | | | |
| 25 Feb 2016 10:45:16 | | | | |
| Line | As 193.759 | Cd 226.502 | Hg 194.227 | Pb 220.353 |
| Conc. | 0.06295 | -0.01925 | 0.04484 | 0.08028 |
| Line | Fe 259.940 | Ni 231.604 | Se 196.090 | Cl 134.724 x |
| Conc. | -0.04927 | -0.27383 | -0.02669 | 79.35902 |
| QC1 - 1 | | | | |
| 25 Feb 2016 11:16:57 | | | | |
| Line | As 193.759 | Cd 226.502 | Hg 194.227 | Pb 220.353 |
| Conc. | 0.03196 | 0.49721 | 0.29952 | 0.09788 |
| Line | Fe 259.940 | Ni 231.604 | Se 196.090 | Cl 134.724 x |
| Conc. | 7.95226 | 9.95527 | 12.01545 | 52.57478 |
| 2/25/16 #6 - 1 | | | | |



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| essed & Understood by me, | Date | Invented by: | Date |
|---|---|---|---|
| | 2/27/16 | Recorded by: | 2/25/16 |

**Giles Chemical, a division of Premier Magnesia, LLC.**
**102 Commerce Street Waynesville, NC 28786**
**Phone: 828-452-4784 Fax: 828-452-4786**

**USP Certificate of Analysis**

Release Date: 2/27/2016

CUSTOMER :

CUST. REF: SHIP DATE:

PRODUCT: Magnesium Sulfate - **USP** TRAILER NUMBER:

SHELF LIFE: 3 Years BILL OF LADING #:

LOT NUMBER: **0916** PO NUMBER #:

CARRIER: MANUFACTURED:

CUST. CONTACT: EXPIRATION:

I certify that this material meets all the requirements of the Giles Chemical sales specifications and the United States Pharmacopeia. Periodic testing has shown the samples to be consistently free of volatile organic impurities. Giles Chemical magnesium sulfate is manufactured under the guidelines of Current Good Manufacturing Practices (cGMP).

**EPSOM SALT, Magnesium Sulfate - Heptahydrate, USP**

| METHOD | TEST | LOWER | UPPER | Test Results |
|---|---|---|---|---|
| VISUAL | Appearance | ~~ | ~~ | Clear |
| USP 38 | Limit of Chloride <221> | ~~ | 140 ppm | < 140 ppm |
| USP 38 | Limit of Iron <241> | ~~ | 20 ppm | < 20 ppm |
| USP 38 | Selenium <291> | ~~ | 30 ppm | < 30 ppm |
| USP 38 | Heavy Metals (as Pb) <231> | ~~ | 10 ppm | < 10 ppm |
| USP 38 | pH (5% Solution) <791> | 5.0 | 9.2 | 7.35 |
| USP 38 | Loss on Ignition <733> | 40.0 | 52.0 | 50.73% |
| USP 38 | Assay, % MgSO4 (Dry Basis) | 99.0 | 100.5 | 99.63% |
| Calculated from Assay & LOI | % MgSO4 (As is Basis) | 47.52 | 60.3 | 49.09% |
| USP 38 | ID, Magnesium <191> | ~~ | ~~ | Present |
| USP 38 | ID, Sulfate <191> | ~~ | ~~ | Present |

CONTACT:

Quality Assurance Laboratory

John Safi - Chemist

828-452-4784 ext. 31

ANALYST:

SIGNATURE ON FILE