
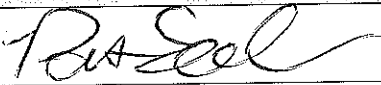

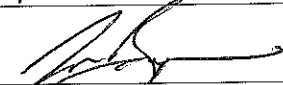
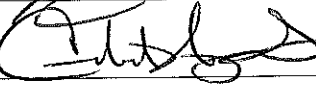

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### Approvals

Signing below indicates agreement that the execution of the Installation, Operational, and Performance Qualification Protocol for Crystallizer #4 located at 102 Commerce Street, Waynesville, NC at the Manufacturing facility is complete and the process is validated.

Project Team Member	Functional Area	Signature	Date
Patrick Owen	Engineering		8/5/13
Robert Willis	Maintenance		8/5/13
Jason Bumgarner	Production		8-5-13
Matt Haynes	Operations		8-5-13
Deborah Durbin	Quality		8-15-13

A copy of the executed protocol will be attached behind the report.

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## I. PURPOSE:

The purpose of this report is to certify with documented evidence that Crystallizer #4 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 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 Crystallizer #4 located in the Manufacturing Building at 102 Commerce Street in Waynesville, NC were all executed and all acceptance criteria were met.

## II. SUMMARY:

A Giles Authorization for Expenditure (AFE) was signed on December 27, 2012 to purchase and install Crystallizer #4 by May 1, 2013. Crystallizer #4 is located in the Manufacturing Building at 102 Commerce Street in Waynesville, NC. 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 impacted by this study were all Epsom Salt products manufactured by Giles Chemical.

The following tests were performed in this qualification:

Each item of Plumbing, Instrumentation, and Utilities was verified to be in place and allow for the specific function as outlined in the Installation Qualification.

It was verified 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.

During the time of the Performance Qualification:

The Vacuum absolute pressure did not exceed 1.50 inches of Mercury in any observation

Fines Reduction Loop flow was not less than 15.0 gallons per minute in any observation

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


Discharge flow was not outside the range of 19-25 gallons per minute in any observation


USP testing of final product passed in all parameters

## III. CONCLUSION

The results of the completed Installation Qualification (IQ), Operational Qualification (OQ), and Performance Qualification (PQ) protocol show that all the acceptance criteria have been met for all samples. All testing results provide documented evidence Crystallizer #4 located in the Manufacturing Building at 102 Commerce Street in Waynesville, NC is installed, operating, and performing as expected. Crystallizer #4 is considered to be validated.

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#### IV. RECOMMENDATIONS

- A. It is recommended that Crystallizer #4 located in the Manufacturing Building at 102 Commerce Street in Waynesville, NC be considered validated based on meeting the acceptance criteria of the IQ/OQ/PQ protocol.

#### V. REFERENCE:

*E35-VAL-PIQ-301, Crystallizer #4 IQ/OQ/PQ Protocol, rev 0, 05/24/13*

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**APPENDIX I: INSTALLATION QUALIFICATION****A. Installation Qualification****1. Location**

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

**2. Level**

Verify that the instrument is level

<b>Is the unit level? (Yes/No)</b>	<b>Acceptable (Yes/No)</b>
YES	YES

**3. Vessel**

<b>Criterion</b>	<b>Are all hatches and outlets tightened, plumbed, or blanked (Yes/No)</b>
Ensure all hatches and outlets are tightened, plumbed, or blanked	YES

**4. Plumbing**

<b>Specified Location</b>	<b>Plumbing present and connected to proper inlet and outlet (Yes/No)</b>
Elbow Pump plumbing	YES
Brine Feed Line	YES
Fine Salt Loop Lines	YES
Discharge Line	YES
Mother Liquor Line	YES
Large Condenser Lines	YES

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Small Condenser Lines	YES
Vacuum Pump plumbing	YES
Ejector Steam Line	YES
Vapor Pipe	YES
Vent Pipe	YES

**5. Instrumentation**

Specified Location	Instrument present and oriented properly (Yes/No)	Instrument Serial Number
Discharge Flow Meter	YES	H300D016000
Mother Liquor Flow Meter	YES	H102F916000
Brine Feed Flow Meter	YES	H102F816000
Fine Salt Loop Flow Meter	YES	H101F316000
Density Transmitter	YES	H101E71509C
Level Transmitter	YES	H101E81509C
Vacuum Transmitter	YES	H101E61509C



**6. Utilities**

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

Specified	Actual
460 V +/- 20V for Pump Drive Panel	474.5
115V +/- 10 for Controls	119.7
Air Present for Vacuum Control Valve	YES
Air Present for Fine Salt Loop Control Valve	YES
Steam Supply for Ejector	YES
Fine Salt Loop Steam	YES

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## APPENDIX II: OPERATIONAL QUALIFICATION

### B. Operational Qualification

#### 1. Vessel and Circulation

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

#### 2. Feed System

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

#### 3. Vacuum System

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

**4. Discharge System**

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



**5. Fine Salt Reduction System**

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

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### 6. Mother Liquor System

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

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**APPENDIX III: PERFORMANCE QUALIFICATION****C. Performance Qualification****1. Vacuum**

<b>Time</b>	<b>Vacuum (&lt; or = 1.50 inches Hg)</b>	<b>Did Item Meet Criteria (Yes/No)</b>
13:00	1.10	YES
13:30	1.10	YES
13:59	1.10	YES
14:30	1.10	YES
15:01	1.08	YES
15:31	1.10	YES
16:00	1.10	YES
16:28	1.10	YES
17:00	1.10	YES

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**2. Fines Reduction Loop Flow**

<b>Time</b>	<b>Fines Reduction Loop Flow (Not Less than 15.0 gallons per minute)</b>	<b>Did Item Meet Criteria (Yes/No)</b>
13:00	17.2	YES
13:30	17.4	YES
13:59	17.4	YES
14:30	17.2	YES
15:01	16.9	YES
15:31	17.2	YES
16:00	17.2	YES
16:28	17.0	YES
17:00	17.2	YES

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**PREMIER**  
MAGNESIA, LLC**3. Fines Reduction Loop Temperature**

<b>Time</b>	<b>Fines Reduction Loop Temperature (38.0 – 44.0 Degrees C)</b>	<b>Did Item Meet Criteria (Yes/No)</b>
13:00	41.0	YES
13:30	41.8	YES
13:59	42.6	YES
14:30	41.8	YES
15:01	41.9	YES
15:31	42.4	YES
16:00	41.0	YES
16:28	41.3	YES
17:00	41.8	YES

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**PREMIER**  
MAGNESIA, LLC**4. Discharge Flow**

Time	Discharge Flow (19 – 25 gallons per minute)	Did Item Meet Criteria (Yes/No)
13:00	21.0	YES
13:30	21.1	YES
13:59	21.0	YES
14:30	21.0	YES
15:01	21.0	YES
15:31	21.2	YES
16:00	21.1	YES
16:28	21.1	YES
17:00	21.0	YES


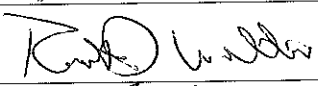
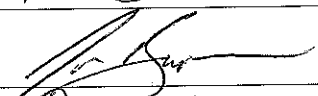
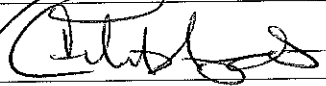
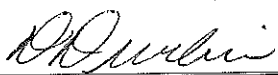
**5. USP Testing**Date and Time Sample Taken : 07:00 8/03/2013**Results: PASS****Note: USP Testing Results attached****Controlled Document**

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#### 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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#### 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.

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- 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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❖ 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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- 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.

### Controlled Document

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## 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

Controlled Document

	<b>GILES CHEMICAL ~ PREMIER MAGNESIA</b>		
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- 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**

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### 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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## 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	Yes	per	8/1/13
<b>Comments:</b> _____			

per  
8/1/13

#### 2. Level

Verify that the instrument is level

LEVEL			
Is the unit level? (Yes/No)	Acceptable (Yes/No)	Verified By	Date
Yes	Yes	per	8/1/13
<b>Comments:</b> _____			

per  
8/1/13

#### 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	Yes	per	8/1/13
<b>Comments:</b> _____			

per  
8/1/13

Reviewed By: Sherry Williams Date: 8/6/13

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## 4. Plumbing

Plumbing			
Specified Location	Plumbing present and connected to proper inlet and outlet (Yes/No)	Verified By	Date
Elbow Pump plumbing	Yes	per	8/1/13
Brine Feed Line	Yes	per	8/1/13
Fine Salt Loop Lines	Yes	per	8/1/13
Discharge Line	Yes	per	8/1/13
Mother Liquor Line	Yes	per	8/1/13
Large Condenser Lines	Yes	per	8/1/13
Small Condenser Lines	Yes	per	8/1/13
Vacuum Pump plumbing	Yes	per	8/1/13
Ejector Steam Line	Yes	per	8/1/13
Vapor Pipe	Yes	per	8/1/13
Vent Pipe	Yes	per	8/1/13
Comments:			

8/1/13  
per

Reviewed By:

Ashley Williams

Date:

8/16/13

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## GILES CHEMICAL ~ PREMIER MAGNESIA

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MAGNESIA, LLC

## 5. Instrumentation

## Instrumentation

Specified Location	Instrument present and oriented properly (Yes/No)	Instrument Serial Number	Verified By	Date
Discharge Flow Meter	YES	11300D016000	per	8/1/13
Mother Liquor Flow Meter	YES	H102F916000	per	8/1/13
Brine Feed Flow Meter	YES	H102F816000	per	8/1/13
Fine Salt Loop Flow Meter	YES	H101F316000	per	8/1/13
Density Transmitter	YES	H101G71509C	per	8/1/13
Level Transmitter	YES	H101E81509C	per	8/1/13
Vacuum Transmitter	YES	H101E61509C	per	8/1/13

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	474.5V	per	8/1/13
115V +/- 10 for Controls	119.7V	per	8/1/13

## Air

Air Present for Vacuum Control Valve	YES	per	8/1/13
Air Present for Fine Salt Loop Control Valve	YES	per	8/1/13

## Steam

Steam Supply for Ejector	YES	per	8/1/13
Fine Salt Loop Steam	YES	per	8/1/13

Comments:

Reviewed By:

Ashley Williams

Date:

8/6/13

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

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**PREMIER**  
MAGNESIA, LLC**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"	Yes	PO	8/1/13
Circulation	With vessel filled with brine and Elbow Pump on circulation discharge should be visible from the top sight glass	Yes	PO	8/1/13
Comments:				

PO  
8/1/13**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	Yes	PO	8/1/13
Brine Pump	With Brine Pump on the motor should turn clockwise as viewed from the motor grill when the drive is "on"	Yes	PO	8/1/13
Brine Flow Meter	With valves open and pump on the Brine Flow Meter on the Brine Flow Meter should register a flow	Yes	PO	8/1/13
Comments:				

PO  
8/1/13

Reviewed By:

Date:

8/6/13

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**PREMIER**  
MAGNESIA, LLC

## 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%	Yes	Per	8/1/13
Vacuum System Utilities	Ensure steam is on Ejector and Water is on both Condensers	Yes	Per	8/1/13
Vacuum Sensor	When Vacuum System is on, the Vacuum Sensor should show the pressure dropping	Yes	Per	8/1/13
Comments:				

Per  
8/1/13

## 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"	Yes	Per	8/1/13
Discharge Flow	With vessel full, open both valves and put system set point on 22 gpm. Verify flow at Centrifuge.	Yes	Per	8/1/13
Comments:				

Per  
8/1/13

Reviewed By:



Ashley Williams

Date:

8/6/13

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### 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%	Yes	PO	8/1/13
Fine Salt Flow	Ensure flow meter shows flow with FS Loop pump on	Yes	POV	8/1/13
Steam Control	Ensure temperature in Fine Salt Loop rises when steam is called for	Yes	POV	8/1/13
Comments:				

POV  
8/1/13

### 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	Yes	POV	8/1/13
Flow	Ensure that when valves are open that flow is observed on the Flow Meter	Yes	POV	8/1/13
Comments:				

POV  
8/1/13

Reviewed By:

*Ashley Williams*

Date:

8/6/13

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## GILES CHEMICAL ~ PREMIER MAGNESIA

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## 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
1300	1.10	yes	POW	8/2/13
1330	1.10	yes	POW	8/2/13
1359	1.10	yes	POW	8/2/13
1430	1.10	yes	POW	8/2/13
1501	1.08	yes	POW	8/2/13
1531	1.10	yes	POW	8/2/13
1600	1.10	yes	POW	8/2/13
1628	1.10	yes	POW	8/2/13
1700	1.10	yes	POW	8/2/13
Comments:				

PO  
8/2/13

Reviewed By:

Ashley Williams

Date:

8/6/13

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## GILES CHEMICAL ~ PREMIER MAGNESIA

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## 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
1300	17.2	Yes	per	8/2/13
1330	17.4	Yes	per	8/2/13
1359	17.4	Yes	per	8/2/13
1430	17.2	Yes	per	8/2/13
1501	16.9	Yes	per	8/2/13
1531	17.2	Yes	per	8/2/13
1600	17.2	Yes	per	8/2/13
1628	17.0	Yes	per	8/2/13
1700	17.2	Yes	per	8/2/13
Comments:				

Reviewed By:

Ashley Williams

Date:

8/6/13

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## 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
1300	41.0	yes	per	8/2/13
1330	41.8	yes	per	8/2/13
1359	42.6	yes	per	8/2/13
1430	41.8	yes	per	8/2/13
1501	41.9	yes	per	8/2/13
1531	42.4	yes	per	8/2/13
1600	41.0	yes	per	8/2/13
1628	41.3	yes	per	8/2/13
1700	41.8	yes	per	8/2/13
Comments:				

Reviewed By:

Ashley Williams

Date:

8/6/13

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## 4. Discharge Flow

Performance Qualification Data				
Time	Discharge Flow (19 – 25 gallons per minute)	Did Item Meet Criteria (Yes/No)	Verified By	Date
1300	21.0	YES	POW	8/2/13
1330	21.1	YES	POW	8/2/13
1359	21.0	YES	POW	8/2/13
1430	21.0	YES	POW	8/2/13
1501	21.0	YES	POW	8/2/13
1531	21.2	YES	POW	8/2/13
1600	21.1	YES	POW	8/2/13
1628	21.1	YES	POW	8/2/13
1700	21.0	YES	POW	8/2/13
Comments:				

POW  
8/2/13

## 5. USP Testing

Date and Time Sample Taken : 0700 8/3/13 Verified By: POW

Results: PASS / FAIL (circle one) Verified By: POW Date: 8/5/13

Note: Attach USP Testing Results to the Final Report

Reviewed By:

Ashley Williams

Date:

8/6/13

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## VII. CALIBRATION VERIFICATION

Equipment	Manufacturer	Model Number	Serial Number	Calibration Date	Verified By	Date
Multimeter	Ideal	61-340	100100221	at manufacturer	Dej	8/1/13
Discharge Flow Meter	Endress + Hauser	Promag 53-P	H300 D01600	3/6/13	Dej	8/1/13
Brine Feed Flow Meter	Endress + Hauser	Promag 53P	H102F816000	1/14/13	Dej	8/1/13
Level Probe	Endress + Hauser	Cerabar S PM71	H101E81509C	1/15/13	Dej	8/1/13
Vacuum Probe	Endress + Hauser	Cerabar S PM71	H101E61509C	1/15/13	Dej	8/1/13
Fines Reduction Flow Meter	Endress + Hauser	Promag 83S	H101F36000	1/14/13	Dej	8/1/13

Reviewed By:

Ashley Williams

Date:

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**ATTACHMENT II. PROTOCOL DEVIATION REPORT (PDR)**

## General Information

System Name: \_\_\_\_\_ Protocol Number: \_\_\_\_\_

Deviation Report Number: \_\_\_\_\_ Protocol Step &amp; 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: \_\_\_\_\_

PDR  
8/2/13

Overall Investigation Review: \_\_\_\_\_

Prepared By: \_\_\_\_\_ Date: \_\_\_\_\_

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## Validation Protocol

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### 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	Engineering	<i>Patrick Owen</i>	PO	8/1/13
Ashley Williams	Quality	<i>Ashley Williams</i>	AW	8/6/13

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Giles Chemical, a division of Premier Magnesia, LLC.

102 Commerce Street Waynesville, NC 28786

USP Certificate of Analysis

Release Date: 8/5/2013

CUSTOMER: \_\_\_\_\_

CUST. REF: \_\_\_\_\_ SHIP DATE: \_\_\_\_\_

PRODUCT: Magnesium Sulfate - USP TRAILER NUMBER: \_\_\_\_\_

SHELF LIFE: 3 Years BILL OF LADING #: \_\_\_\_\_

LOT NUMBER: 1913 PO NUMBER #: \_\_\_\_\_

SHIPPER: \_\_\_\_\_ MANUFACTURED: \_\_\_\_\_

CUST. SERV.: \_\_\_\_\_ EXPIRATION: \_\_\_\_\_

I certify that this material meets all the requirements of the Giles Chemical sales specifications and the United States Pharmacopeia 35. 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
USP35	Chloride as Cl (%)	~~	0.014	< 0.014%
USP35	Heavy Metals As Pb (%)	~~	0.001	< 0.001%
USP35	Ignition Loss (%)	40.00	52.00	51.23%
USP35	Iron (ppm)	~~	20.00	< 20.00 ppm
USP35	MgSO <sub>4</sub> ( Anhydrous wt. % )	99.00	100.50	99.90%
USP35	MgSO <sub>4</sub> ( As is basis, % )	48.30	52.00	48.75%
USP35	pH 5% Solution	5.00	9.20	7.80
USP35	Selenium (%)	~~	0.003	< 0.003%
USP35	Mg <sup>2+</sup> ID			Present
USP35	SO <sub>4</sub> <sup>2-</sup> ID			Present

ATTN:

ANALYST:

SIGNATURE ON FILE

## Flow Calibration with Adjustment

30263727-2982132

1000219296

Purchase order number

US-3004970133-10 / Endress+Hauser Flowtec

Order N°/Manufacturer

53P40-EL2B1AA0BAAA

Order code

PROMAG 53 P 1 1/2"

Transmitter/Sensor

H300D016000

Serial N°

-

Tag N°

FCP-6.C

Calibration rig

99.59059 us.gal/min ( $\pm 100\%$ )

Calibrated full scale

Service interface

Calibrated output

0.7625

Calibration factor

-10

Zero point

82 °F

Water temperature

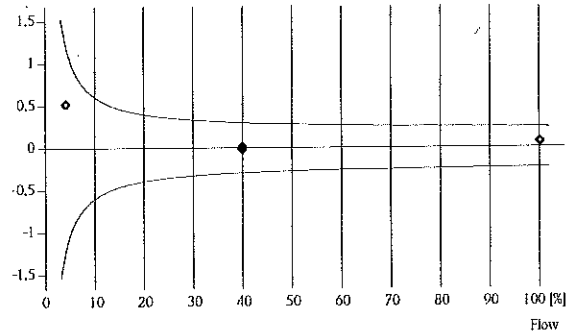
Flow [%]	Flow [us.gal/min]	Duration [s]	V target [us.gal]	V meas. [us.gal]	$\Delta$ o.r.* [%]	Outp.** [mA]
4.0	3.94	85.1	5.5924	5.6222	0.53	4.64
39.9	39.8	60.1	39.862	39.856	-0.01	10.39
40.0	39.8	60.2	39.926	39.931	0.01	10.40
100.2	99.8	50.2	83.424	83.483	0.07	20.04
-	-	-	-	-	-	-
-	-	-	-	-	-	-
-	-	-	-	-	-	-
-	-	-	-	-	-	-
-	-	-	-	-	-	-
-	-	-	-	-	-	-

\*o.r.: of rate

\*\*Calculated value (4 - 20 mA)

Measured error % o.r.

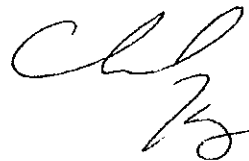
Tolerance limit:  $\pm 0.2\%$  o.r. \*  $\pm$  Zero stability



For detailed data concerning output specifications of the unit under test, see Technical Information (TI), chapter Performance characteristics.

The calibration is traceable to the N.I.S.T. through standards certified at preset intervals.

Endress+Hauser Flowtec operates ISO/IEC 17025 accredited calibration facilities in Reinach (CH), Cernay (FR), Greenwood (USA), Aurangabad (IN) and Suzhou (CN).



Chad Key

Operator

Certified acc. to  
ISO 9001, Reg.-N° 030502.2  
ISO 14001, Reg.-N° EMS561046

03-06-2013

Date of calibration

Endress+Hauser Flowtec, Division USA  
2330 Endress Place  
Greenwood, IN 46143

## Flow Calibration with Adjustment

30258815-2944984

1000214704

Purchase order number

US-3004945709-30 / Endress+Hauser Flowtec

Order N°/Manufacturer

53P40-EL0B1AA0BAAA

Order code

PROMAG 53 P 1 1/2"

Transmitter/Sensor

H102F816000

Serial N°

-

Tag N°

FCP-6.C

Calibration rig

99.59059 us.gal/min ( $\pm 100\%$ )

Calibrated full scale

Service interface

Calibrated output

0.7872

Calibration factor

10

Zero point

76.1 °F

Water temperature

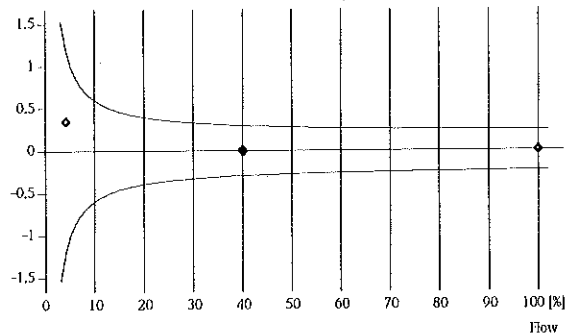
Flow [%]	Flow [us.gal/min]	Duration [s]	V target [us.gal]	V meas. [us.gal]	$\Delta$ o.r.* [%]	Outp.** [mA]
4.0	3.99	85.1	5.6655	5.6858	0.36	4.64
40.0	39.8	60.1	39.903	39.908	0.01	10.40
40.1	39.9	60.1	39.967	39.966	0.00	10.41
100.1	99.6	50.1	83.258	83.268	0.01	20.01
-	-	-	-	-	-	-
-	-	-	-	-	-	-
-	-	-	-	-	-	-
-	-	-	-	-	-	-
-	-	-	-	-	-	-
-	-	-	-	-	-	-

\*o.r.: of rate

\*\*Calculated value (4 - 20 mA)

Measured error % o.r.

Tolerance limit:  $\pm 0.2\%$  o.r.\*  $\pm$  Zero stability



For detailed data concerning output specifications of the unit under test, see Technical Information (TI), chapter Performance characteristics.

The calibration is traceable to the N.I.S.T. through standards certified at preset intervals.

Endress+Hauser Flowtec operates ISO/IEC 17025 accredited calibration facilities in Reinach (CH), Cernay (FR), Greenwood (USA), Aurangabad (IN) and Suzhou (CN).

*David McCombs*

01-14-2013

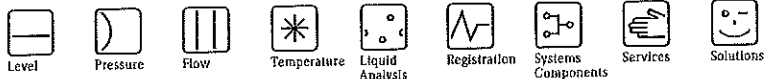
Date of calibration

Endress+Hauser Flowtec, Division USA  
2330 Endress Place  
Greenwood, IN 46143

David McCombs

Operator

Certified acc. to  
ISO 9001, Reg.-N° 030502.2  
ISO 14001, Reg.-N° EMS561046



Endress+Hauser GmbH+Co.  
2340 Endress Place  
Greenwood, In. 46143-9772

## Final Inspection Report / Endprüfprotokoll

The manufacturer confirms that all measuring equipment used to assure the quality of the products has been calibrated and is traceable to national (e.g. DKD/DAkkS, NIST, NABL...) or international standards.

Der Hersteller bestätigt, dass die zu Qualitätsprüfungen des Erzeugnisses eingesetzten Messmittel gültig kalibriert waren und auf nationale (z.B. DKD/DAkkS, NIST, NABL...) bzw. internationale Normale rückführbar sind.

### Cerabar S

TAG number

Messstellen-Nummer

Order code  
Serial number  
Extended order code  
Sensor range  
Adjusted measuring range  
Maximum permissible error  
Output type  
Software version  
Output mode

Bestellcode  
Seriennummer  
Erweiterter Bestellcode  
Sensor-Messbereich  
Eingestellter Messbereich  
Max. zulässige Messabweichung  
Ausgang  
Softwareversion  
Ausgangsmodus

PMC71-SAC2K4AGAAA  
H101E81509C  
PMC71-SAC2K4AGAAA  
0...800 inH<sub>2</sub>O abs  
0...800 inH<sub>2</sub>O abs  
± 0.075 %  
4...20 mA, HART  
02.10.54  
linear

### GILES CHEMICAL

Customer order number  
E+H sales order number  
Internal order number

Auftragsnummer des Kunden  
E+H Auftragsnummer  
Interne Auftragsnummer

129155  
71235087000060  
1000214704/0060

Ambient temperature  
Ambient humidity  
Ambient pressure

Umgebungs-Temperatur  
Umgebungs-Luftfeuchte  
Umgebungs-Luftdruck

22.6 °C (± 1 °C)  
22.7 %rel.F (± 10 %rel.F)  
994.5 mbar (± 0.2 mbar)

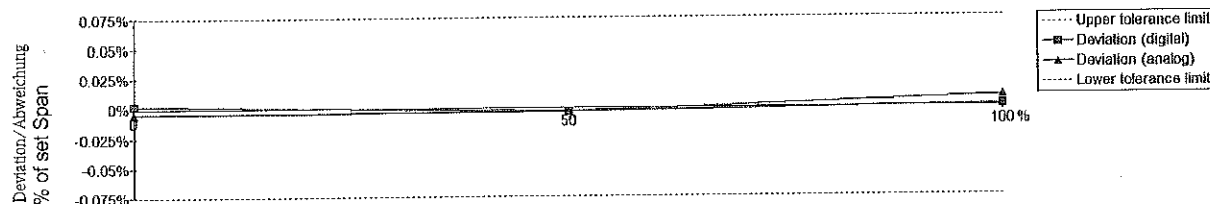
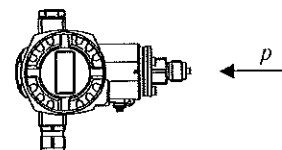
Calibrated according to fix point method IEC 60770.

Prüfung nach Grenzpunktmethode gemäß IEC 60770.

#### Measuring results / Messergebnisse

Calibration orientation  
Kalibrierlage

Calibration point	Nominal value (P Ref.)	Measured value (digital readout)	Deviation (digital)	Nominal value (I <sub>OUT</sub> calculated)	Current output (analog)	Rel. deviation (analog)
Kalibrierpunkt	Sollwert (P Ref.)	Istwert (Digitaler Wert)	Abweichung (digital)	Sollwert (I <sub>OUT</sub> berechnet)	Stromausgang (analog)	Rel. Abweichung (analog)
%	inH <sub>2</sub> O	inH <sub>2</sub> O	% of Span	mA	mA	%
0	12.9640	12.9842	0.00253	4.2593	4.2585	-0.0047
50	399.280	399.254	-0.00327	11.986	11.985	-0.0031
100	806.744	806.751	0.00088	20.135	20.136	0.0090



Calibration carried out in output mode linear /  
Kalibration erfolgte im Ausgangsmodus linear.

Measuring point in % of adjusted measuring range /  
Messpunkt in % vom eingestellten Messbereich

We confirm that all tests, according to the Inspection and Test Plan (ITP), have been performed successfully. At the time of verification, the measuring points of the device indicated above were in compliance to the published valid technical specification (TI).

Wir bestätigen, dass alle Tests aus den Inspektions- und Testplänen (ITP) erfolgreich durchgeführt wurden. Das Gerät entsprach zum Zeitpunkt der Prüfung an den aufgeführten Messpunkten den gültigen technischen Spezifikationen (TI).

TI 383P

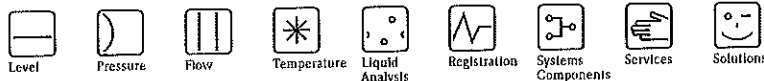
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Operator / geprüft durch 155448  
Date of inspection / Prüfdatum 15. Jan 2013

**Endress+Hauser**   
People for Process Automation





Endress+Hauser GmbH+Co.  
2340 Endress Place  
Greenwood, In. 46143-9772

## Final Inspection Report / Endprüfprotokoll

The manufacturer confirms that all measuring equipment used to assure the quality of the products has been calibrated and is traceable to national (e.g. DKD/DAKKS, NIST, NABL...) or international standards.

Der Hersteller bestätigt, dass die zu Qualitätsprüfungen des Erzeugnisses eingesetzten Messmittel gültig kalibriert waren und auf nationale (z.B. DKD/DAKKS, NIST, NABL...) bzw. internationale Normale rückführbar sind.

### Cerabar S

TAG number

Messstellen-Nummer

Order code	Bestellcode	PMC71-SAC2K4RAAAU
Serial number	Seriennummer	H101E61509C
Extended order code	Erweiterter Bestellcode	PMC71-SAC2K4RAAAU
Sensor range	Sensor-Messbereich	0...800 inH <sub>2</sub> O abs
Adjusted measuring range	Eingestellter Messbereich	0...800 inH <sub>2</sub> O abs
Maximum permissible error	Max. zulässige Messabweichung	± 0.075 %
Output type	Ausgang	4...20 mA, HART
Software version	Softwareversion	02.10.54
Output mode	Ausgangsmodus	linear

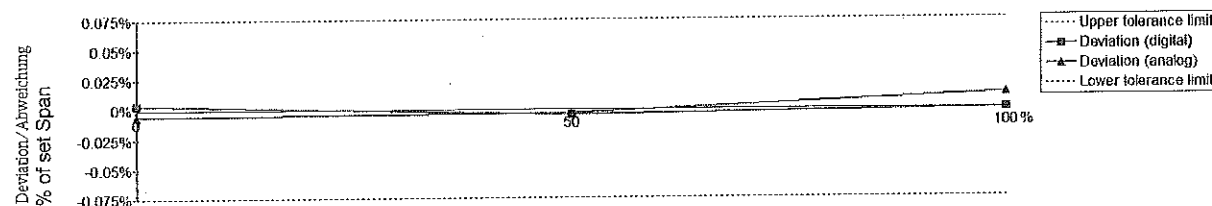
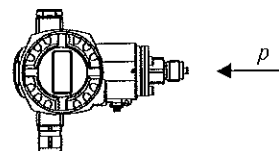
### GILES CHEMICAL

Customer order number	Auftragsnummer des Kunden	129155
E+H sales order number	E+H Auftragsnummer	71235087000050
Internal order number	Interne Auftragsnummer	1000214704/0050
Ambient temperature	Umgebungs-Temperatur	22.4 °C (± 1 °C)
Ambient humidity	Umgebungs-Luftfeuchte	22.4 %rel.F (± 10 %rel.F)
Ambient pressure	Umgebungs-Luftdruck	994.5 mbar (± 0.2 mbar)
Calibrated according to fix point method IEC 60770.	Prüfung nach Grenzpunktmethode gemäß IEC 60770.	

#### Measuring results / Messergebnisse

Calibration point	Nominal value (p Ref.)	Measured value (digital readout)	Deviation (digital)	Nominal value (I <sub>OUT</sub> calculated)	Current output (analog)	Rel. deviation (analog)
Kalibrierpunkt	Sollwert (p Ref.)	Istwert (Digitaler Wert)	Abweichung (digital)	Sollwert (I <sub>OUT</sub> berechnet)	Istwert Stromausgang (analog)	Rel. Abweichung (analog)
%	inH <sub>2</sub> O	inH <sub>2</sub> O	% of Span	mA	mA	%
0	12.3160	12.3479	0.00398	4.2463	4.2454	-0.0055
50	399.228	399.191	-0.00459	11.985	11.984	-0.0036
100	801.456	801.450	-0.00071	20.029	20.031	0.0122

Calibration orientation  
Kalibrierlage



Calibration carried out in output mode linear/  
Kalibration erfolgte im Ausgangsmodus linear.

We confirm that all tests, according to the Inspection and Test Plan (ITP), have been performed successfully. At the time of verification, the measuring points of the device indicated above were in compliance to the published valid technical specification (TI).

TI 383P

Measuring point in % of adjusted measuring range/  
Messpunkt in % vom eingestellten Messbereich

Wir bestätigen, dass alle Tests aus den Inspektions- und Testplänen (ITP) erfolgreich durchgeführt wurden. Das Gerät entsprach zum Zeitpunkt der Prüfung an den aufgeführten Messpunkten den gültigen technischen Spezifikationen (TI).

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Operator /geprüft durch 155448  
Date of inspection / Prüfdatum 15. Jan 2013

**Endress+Hauser**   
People for Process Automation

## Flow Calibration with Adjustment

30258846-2942979

1000214704

Purchase order number

US-3004945709-40 / Endress+Hauser Flowtec

Order N°/Manufacturer

83S25-1020/0

Order code

PROMASS 83 S 1"

Transmitter/Sensor

H101F316000

Serial N°

-

Tag N°

FCP-6.F

Calibration rig

132.2774 lb/min ( $\pm 100\%$ )

Calibrated full scale

Service interface

Calibrated output

3.2299

Calibration factor

24

Zero point

77.1 °F

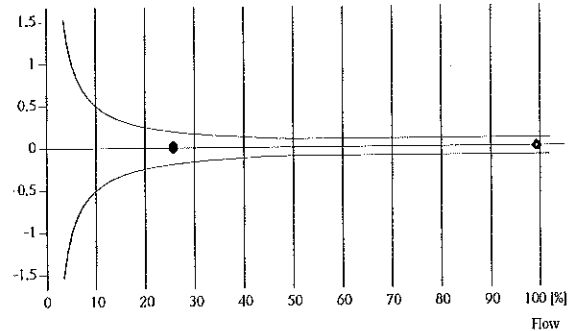
Water temperature

Flow [%]	Flow [lb/min]	Duration [s]	m target [lb]	m meas. [lb]	$\Delta$ o.r.* [%]
25.6	33.9	85.2	48.120	48.135	0.03
25.6	33.9	85.2	48.126	48.124	-0.01
99.4	131.4	30.2	66.139	66.140	0.00
99.5	131.6	30.2	66.205	66.204	0.00
-	-	-	-	-	-
-	-	-	-	-	-
-	-	-	-	-	-
-	-	-	-	-	-
-	-	-	-	-	-
-	-	-	-	-	-

\*o.r.: of rate

Measured error % o.r.

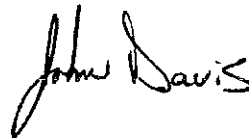
Tolerance limit:  $\pm 0.1\%$  o.r.



For detailed data concerning output specifications of the unit under test, see Technical Information (TI), chapter Performance characteristics.

The calibration is traceable to the N.I.S.T. through standards certified at preset intervals.

Endress+Hauser Flowtec operates ISO/IEC 17025 accredited calibration facilities in Reinach (CH), Cernay (FR), Greenwood (USA), Aurangabad (IN) and Suzhou (CN).



John Davis  
Operator

01-14-2013

Date of calibration

Endress+Hauser Flowtec, Division USA  
2330 Endress Place  
Greenwood, IN 46143

Certified acc. to  
ISO 9001, Reg.-N° 030502.2  
ISO 14001, Reg.-N° EMS561046

## Flow Calibration with Adjustment

30258873-2944985

1000214704

Purchase order number

US-3004945709-20 / Endress+Hauser Flowtec

Order N°/Manufacturer

53P25-EL2B1AA0BAAA

Order code

PROMAG 53 P 1"

Transmitter/Sensor

H102F916000

Serial N°

-

Tag N°

FCP-6.C

Calibration rig

38.90256 us.gal/min ( $\pm 100\%$ )

Calibrated full scale

Service interface

Calibrated output

0.7457

Calibration factor

0

Zero point

75.6 °F

Water temperature

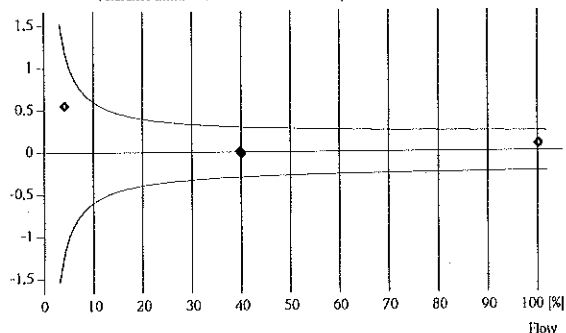
Flow [%]	Flow [us.gal./min]	Duration [s]	V target [us.gal]	V meas. [us.gal]	$\Delta$ o.r.* [%]	Outp.** [mA]
4.0	1.56	60.1	1.5642	1.5731	0.57	4.65
39.8	15.5	60.1	15.524	15.526	0.01	10.37
40.0	15.6	60.1	15.601	15.599	-0.01	10.40
100.3	39.0	60.1	39.103	39.138	0.09	20.06
-	-	-	-	-	-	-
-	-	-	-	-	-	-
-	-	-	-	-	-	-
-	-	-	-	-	-	-
-	-	-	-	-	-	-
-	-	-	-	-	-	-

\*o.r.: of rate

\*\*Calculated value (4 - 20 mA)

Measured error % o.r.

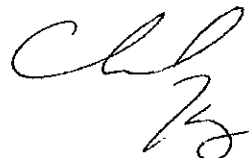
Tolerance limit:  $\pm 0.2\%$  o.r.\*  $\pm$  Zero stability



For detailed data concerning output specifications of the unit under test, see Technical Information (TI), chapter Performance characteristics.

The calibration is traceable to the N.I.S.T. through standards certified at preset intervals.

Endress+Hauser Flowtec operates ISO/IEC 17025 accredited calibration facilities in Reinach (CH), Cernay (FR), Greenwood (USA), Aurangabad (IN) and Suzhou (CN).



Chad Key

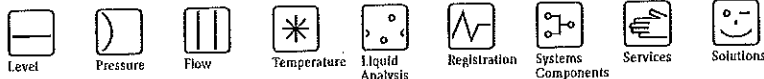
Operator

Certified acc. to  
ISO 9001, Reg.-N° 030502.2  
ISO 14001, Reg.-N° EMS561046

01-14-2013

Date of calibration

Endress+Hauser Flowtec, Division USA  
2330 Endress Place  
Greenwood, IN 46143



Endress+Hauser GmbH+Co.  
2340 Endress Place  
Greenwood, In. 46143-9772

## Final Inspection Report / Endprüfprotokoll

The manufacturer confirms that all measuring equipment used to assure the quality of the products has been calibrated and is traceable to national [e.g. DKD/DAkkS, NIST, NABL....] or international standards.

Der Hersteller bestätigt, dass die zu Qualitätsprüfungen des Erzeugnisses eingesetzten Messmittel gültig kalibriert waren und auf nationale (z.B. DKD/DAkkS, NIST, NABL....) bzw. internationale Normale rückführbar sind.

### Cerabar S

TAG number

Messstellen-Nummer

Order code  
Serial number  
Extended order code  
Sensor range  
Adjusted measuring range  
Maximum permissible error  
Output type  
Software version  
Output mode

Bestellcode  
Seriennummer  
Erweiterter Bestellcode  
Sensor-Messbereich  
Eingestellter Messbereich  
Max. zulässige Messabweichung  
Ausgang  
Softwareversion  
Ausgangsmodus

PMC71-SAC2K4GAAAA  
H101E71509C  
PMC71-SAC2K4GAAAA  
0...800 inH<sub>2</sub>O abs  
0...800 inH<sub>2</sub>O abs  
± 0.075 %  
4...20 mA, HART  
02.10.54  
linear

### GILES CHEMICAL

Customer order number  
E+H sales order number  
Internal order number

Auftragsnummer des Kunden  
E+H Auftragsnummer  
Interne Auftragsnummer

129155  
71235087000060  
1000214704/0060

Ambient temperature  
Ambient humidity  
Ambient pressure

Umgebungs-Temperatur  
Umgebungs-Luftfeuchte  
Umgebungs-Luftdruck

22.6 °C (± 1 °C)  
22.3 %rel.F (± 10 %rel.F)  
993.8 mbar (± 0.2 mbar)

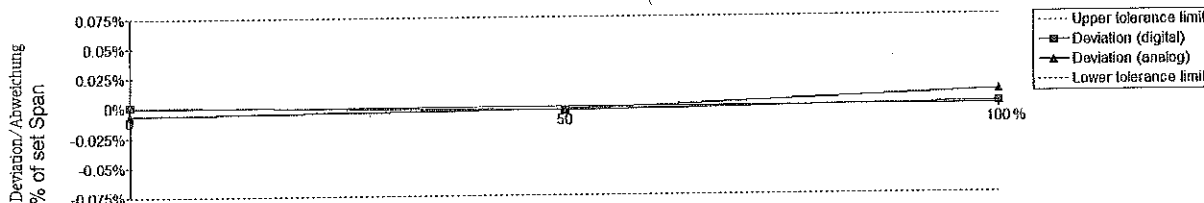
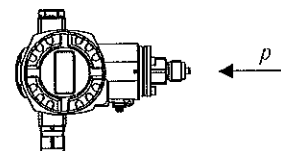
Calibrated according to fix point method IEC 60770.

Prüfung nach Grenzpunktmethode gemäß IEC 60770.

#### Measuring results / Messergebnisse

Calibration orientation  
Kalibrierrichtung

Calibration point	Nominal value (P Ref.)	Measured value (digital readout)	Deviation (digital)	Nominal value (I <sub>Out</sub> calculated)	Current output (analog)	Rel. deviation (analog)
Kalibrierpunkt	Sollwert (P Ref.)	Istwert (Digitaler Wert)	Abweichung (digital)	Sollwert (I <sub>Out</sub> berechnet)	Istwert Stromausgang (analog)	Rel. Abweichung (analog)
%	inH <sub>2</sub> O	inH <sub>2</sub> O	% of Span	mA	mA	%
0	12.3400	12.3519	0.00149	4.2468	4.2458	-0.0061
50	399.000	398.975	-0.00316	11.980	11.980	-0.0028
100	804.200	804.217	0.00216	20.084	20.086	0.0118



Calibration carried out in output mode linear/  
Kalibration erfolgte im Ausgangsmodus linear.

Measuring point in % of adjusted measuring range/  
Messpunkt in % vom eingestellten Messbereich

We confirm that all tests, according to the inspection and Test Plan (ITP), have been performed successfully. At the time of verification, the measuring points of the device indicated above were in compliance to the published valid technical specification (TI).

Wir bestätigen, dass alle Tests aus den Inspektions- und Testplänen (ITP) erfolgreich durchgeführt wurden. Das Gerät entsprach zum Zeitpunkt der Prüfung an den aufgeführten Messpunkten den gültigen technischen Spezifikationen (TI).

TI 383P

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Operator / geprüft durch 155448  
Date of inspection / Prüfdatum 15. Jan 2013

**Endress+Hauser**   
People for Process Automation

31065312-2942979-675807

Transmitter/Sensor

H101F316000

Serial N°

FCP-6.F

---

Calibration rig

$$C0 = -2531.3$$

Density coefficient

C1 = 1.5668E+9

C2 = -7.6621E+5

C3 = -36033

Density coefficient

C4 = 1.1054E+14

C5 = -1.2950E-3

Density coefficient

Endress+Hauser Flowtec operates ISO/IEC 17025 accredited calibration facilities in Reinach (CH), Cernay (FR), Greenwood (USA), Aurangabad (IN) and Suzhou (CN).

John Davis

01-14-2013

Date of calibration

Endress+Hauser Flowtec, Division USA  
2330 Endress Place  
Greenwood, IN 46143

John Davis

Operator

Certified acc. to  
ISO 9001, Reg.-N° 030502.2  
ISO 14001, Reg.-N° EMS561046