

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

Title: Digester #2 IQ/OQ/PQ Validation

Number: E17-VAL-PIQ-260

Owner: Kenneth Baschore

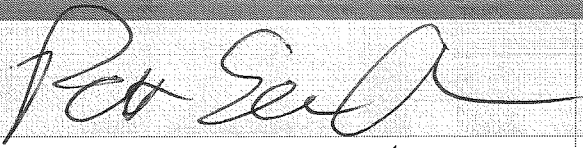

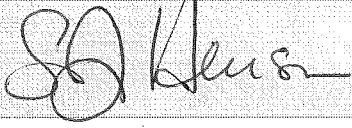
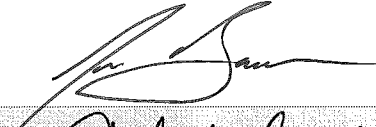
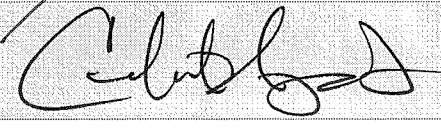

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**I. Approvals**

Signing below indicates agreement that the protocol is ready for execution of the Installation, Operational, and Performance Qualification for the Digester #2, located at 102 Commerce Street at the Main Plant production facility.

Project Member	Functional Area	Signature	Date
Patrick Owen	Engineering		4/5/17
Kenneth Baschore	Engineering		4/5/17
Sammy Henson	Maintenance		4/5/17
Jason Bumgarner	Production		4-5-17
Matt Haynes	Operations		4-5-17
Deborah Durbin	Quality		4/5/17

A final summary report that consists of results and conclusions based on the data collected after protocol execution will be written and approved. The executed protocol will be attached behind the report.

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II. Purpose

The purpose of this protocol is to certify with documented evidence that Digester #2 functions as intended throughout its anticipated operating ranges. This protocol sets forth the objectives, methodology, documentation, and test activities needed to complete the Installation Qualification (IQ), Operational Qualification (OQ) and Process Qualification (PQ) for Digester #2, located at 102 Commerce Street at the Main Plant production facility.

III. Background

Digester #2 was custom built on site by contractors. It is intended to receive an MgO slurry from the Main MgO Mix Pot, sulfuric acid from the acid storage tanks, city water and liquor (a by-product of centrifugation). The resultant mix is allowed to react and form a slurry of dissolved MgSO₄, natural minerals and water. The slurry ('mud') is overflowed into a secondary digester for further dwell time, which is then overflowed to storage tanks.

IV. Overview

No other departments or systems will be affected by the installation of use or this equipment.

The following tests will be performed during this qualification:

- Reagent feed to the digester
- Reaction exotherm
- Agitator run state
- Recirculation flow rate

V. System Description

1. Digester #2 is turned on through the Monitoring System
2. Once the pH drops below 5.0, the monitoring system will maintain a pH setpoint, defined by the operator
3. The recirculation loop must maintain at least 50 gpm, or the digester will shut down
4. The primary digester overflows into the secondary digester
5. The secondary digester overflows into the mud storage tanks

VI. Scope

The IQ, OQ and PQ contained within this protocol is intended to certify with documented evidence that Digester #2 is installed, operates and functions as intended throughout its anticipated operating ranges.

The product affected by this equipment is all salt produced in the Main Plant at 102 Commerce Street, Waynesville, NC.

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VII. Roles and Responsibilities

1. Engineering

- Write and issue the protocol
- Investigate protocol deviation reports
- Execute the IQ, OQ and PQ portions of the validation
- Review the data and originate the interim notification to Quality Assurance
- Write and route the final report

2. Quality Assurance

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

3. Maintenance

- Provide equipment manuals needed to execute the validation
- Review and approve the protocol
- Review and approve the raw data and notifications
- Review and approve the final report

4. Production

- Review and approve the protocol
- Review and approve the raw data and notifications
- Review and approve the final report
- Assist, as needed with the execution of the IQ, OQ and PQ

VIII. Test Program

1. Installation Qualification (IQ)

a. Objective

The objective of the installation verification is to document that Digester #2 is installed correctly.

b. Equipment and Materials

- Digester #2
- Agitator
- Recirculation Flow Meter
- Recirculation Pump

c. Procedure

- Verify that the equipment is situated to allow sufficient room around the machine for access.
- Verify that the equipment is level
- Verify that the electrical utilities fall within the manufacturers required ranges

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d. Acceptance Criteria

Ensure that the installation is correct, per the design drawings.

2. Operational Qualification (OQ)

a. Objective

The objective of the operational qualification is to ensure that Digester #2 operates as indicated by the design drawings. The controls will be operated to test the ability of the vessel to start and stop as the circulation flow rate varies above and below the alarm limits.

b. Equipment and Materials

Digester #2

c. Procedure

Test each operation of Digester #2

d. Acceptance Criteria

Verification that the tested operations operate as indicated by the designer's specifications.

3. Performance Qualification (PQ)

a. Objective

The objective of the performance testing is to document that Digester #2 performs the functions required by Giles Chemical. This protocol will verify the following:

- The Digester temperature varies with MgO feed
- The acid charge varies with pH setpoint variation
- The agitator continues to run

b. Equipment and Materials

- Digester #2
- Agitator

c. Procedure

Run the machine for long enough to allow the feed rates to equilibrate. Verify that the agitator is running. Reduce the MgO feed rate, and verify that the temperature drops. Change the pH setpoint, and verify that the acid feed changes.

d. Acceptance Criteria

The PQ will be accepted if the MgO feed rate and pH setpoints alter the temperature and acid feed rate of the vessel.

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IX. Calibration

Verify that all instruments used are within the calibration dates.

- Calibrated multimeter

X. References

- N/a

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**XI. Installation Qualification (IQ)****1. Equipment**

Device	Calibration Date	Calibration Expiration	Verified By	Date
Multimeter Model: Fluke 114 S/N: 36250117WS	10/3/16	10/3/17	KLB	9/19/17

Expected	Actual	Verified By	Date
Agitator Model: P18G1133E S/N: P18G133-001	AGITATOR MODEL: P18G1133E S/N: P18G133-001	KLB	9/19/17
Digester #2	DIGESTER #2	KLB	9/19/17
Circulation Pump Model: 2196STO S/N: 335675	CIRCULATION PUMP MODEL: 2196STO S/N: 335675	KLB	9/19/17
Flow Meter Model: 83S50 S/N: L808CF16000	FLOW METER MODEL: 83S50 S/N: L808CF16000	KLB	9/19/17

2. Acceptance Testing

Expected	Actual	Verified By	Date
There is sufficient room around the vessel to allow access doors and panels to be opened.	There <u>IS</u> sufficient room around the machine to allow access doors and panels to be opened.	KLB	9/19/17
The equipment is level	The equipment <u>IS</u> level	KLB	9/19/17
Agitator power supply 240 VAC \pm 20	240 VAC	KLB	9/19/17
Agitator power supply 60 Hz	60 Hz	KLB	9/19/17

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Flow meter power supply 110 VAC \pm 10	110 VAC	KLB	9/19/17
Flow meter power supply 60 Hz	60 Hz	KLB	9/19/17
Circulation pump power supply 230 VAC \pm 20	230 VAC	KLB	9/19/17
Circulation pump power supply 60 Hz	60 Hz	KLB	9/19/17
Acid lines are piped correctly	Acid lines <u>ARE</u> piped correctly	KLB	9/19/17
MgO supply lines are piped correctly	MgO supply lines <u>ARE</u> piped correctly	KLB	9/19/17
Water lines are piped correctly	Water lines <u>ARE</u> piped correctly	KLB	9/19/17
Liquor lines are piped correctly	Liquor lines <u>ARE</u> piped correctly	KLB	9/19/17

3. Acceptance of Testing and Review

Expected	Actual	Initials	Date
All actual results match the expected values.	All actual results <u>MATCH</u> the expected values.	KLB	9/19/17
The relevant standard working procedures are approved	The relevant standard working procedures <u>ARE</u> approved	KLB	9/19/17

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List the procedure numbers

P12-PR-200-013
P17-PR-200-098
P17-PR-200-099
P12-PR-200-015
P12-PR-200-012
P12-PR-200-097

KLB

9/19/17

Results reviewed and accepted by

aw

9/19/17

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XII. Operational Qualification (OQ)

1. Equipment

Expected	Actual	Verified By	Date
Agitator Model: P18G1133E S/N: P18G133-001	AGITATOR MODEL: P18G1133E S/N: P18G133-001	KLB	9/19/17
Digester #2	DIGESTER #2	KLB	9/19/17
Circulation Pump Model: 2196STO S/N: 335675	CIRCULATION PUMP MODEL: 2196STO S/N: 335675	KLB	9/19/17
Flow Meter Model: 83S50 S/N: L808CF16000	FLOW METER MODEL: 83S50 S/N: L808CF16000	KLB	9/19/17

2. Acceptance Testing

Expected	Actual	Verified By	Date
The vessel is stopped	The vessel <u>IS</u> stopped	KLB	9/19/17
The agitator is stopped	The agitator <u>IS</u> stopped	KLB	9/19/17
Press the start button to start the agitator	The agitator <u>IS</u> started	KLB	9/19/17
The agitator is running	The agitator <u>IS</u> running	KLB	9/19/17
Press the "Start Digester" button in the monitoring system	The button <u>IS</u> pressed	KLB	9/19/17
The circulation loop is running	The circulation loop <u>IS</u> running	KLB	9/19/17

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Record the circulation loop flow rate	Flow rate: <u>126 GPM</u>	KLB	9/19/17
Lower the flow rate to below 50 gpm	New flow rate: <u>49 GPM</u>	KLB	9/19/17
The digester shut down	The digester <u>SHUT</u> down	KLB	9/19/17
Reset the flow rate to the original value	Flow rate: <u>126 GPM</u>	KLB	9/19/17
Press the "Start Digester" button in the monitoring system	The button <u>IS</u> pressed	KLB	9/19/17
The circulation loop is running	The circulation loop <u>IS</u> running	KLB	9/19/17
Lower the pH setpoint to below the current value	Current pH: <u>3.7</u> New setpoint: <u>3.0</u>	KLB	9/19/17
The acid flow starts	The acid flow <u>STARTS.</u>	KLB	9/19/17
Raise the pH setpoint to above the current value	Current pH: <u>3.7</u> New setpoint: <u>4.0</u>	KLB	9/19/17
The acid flow stops	The acid flow <u>STOPS.</u>	KLB	9/19/17
Return the pH setpoint to the original value	pH setpoint: <u>3.8</u>	KLB	9/19/17
Stop the agitator	The agitator <u>IS</u> stopped	KLB	9/19/17

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The digester shuts down

The digester SHUTS down

KLB

9/19/17

3. Acceptance of Testing and Review

Expected	Actual	Initials	Date
All actual results match the expected values.	All actual results <u>MATCH</u> the expected values.	KLB	9/19/17
The IQ section is complete with no deviations	The IQ section <u>IS</u> complete with no deviations	KLB	9/19/17
Results reviewed and accepted by		aw	9/19/17

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XIII. Performance Qualification (PQ)

1. Equipment

Expected	Actual	Verified By	Date
Agitator Model: P18G1133E S/N: P18G133-001	AGITATOR MODEL: P18G1133E S/N: P18G133-001	KLB	9/20/17
Digester #2	DIGESTER #2	KLB	9/20/17
Circulation Pump Model: 2196STO S/N: 335675	CIRCULATION PUMP MODEL: 2196STO S/N: 335675	KLB	9/20/17
Flow Meter Model: 83S50 S/N: L808CF16000	FLOW METER MODEL: 83S50 S/N: L808CF16000	KLB	9/20/17

2. Acceptance Testing

Expected	Actual	Initials	Date
Press the start button to start the agitator	The agitator <u>15</u> started	KLB	9/20/17
The agitator is running	The agitator <u>15</u> running	KLB	9/20/17
Press the "Start Digester" button in the monitoring system	The button <u>15</u> pressed	KLB	9/20/17
The circulation loop is running	The circulation loop <u>15</u> running	KLB	9/20/17
Allow the digester to operate undisturbed for 1 hour	Start time: 0730 End time: 0830	KLB	9/20/17
Stop the Main MgO Mix Pot	The Main MgO Mix Pot is <u>STOPPED</u> .	KLB	9/20/17

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Within 30 minutes, Digester #2's temperature drops at least 5 degrees	Start time and temperature: 8:45 99.5°C End time and temperature: 9:15 94.3°C	KLB	9/20/17
Turn on the Main MgO Mix Pot	The Main MgO Mix Pot <u>is</u> on	KLB	9/20/17
Allow the digester to operate undisturbed for 1 hour	Start time: 9:20 End time: 10:20	KLB	9/20/17
Manually close the acid line shutoff valve	The valve <u>is</u> closed	KLB	9/20/17
Within 30 minutes, Digester #2's temperature drops at least 5 degrees	Start time and temperature: 10:25 98.7°C End time and temperature: 10:50 93.0°C	KLB	9/20/17
Manually open the acid line shutoff valve	The valve <u>is</u> open	KLB	9/20/17
Allow the digester to operate undisturbed for 1 hour	Start time: 10:55 End time: 11:55	KLB	9/20/17
Manually close the liquor line shutoff valve	The valve <u>is</u> closed	KLB	9/20/17
Within 30 minutes, Digester #2's temperature increases at least 5 degrees	Start time and temperature: 12:00 91.1°C End time and temperature: 12:27 98.2°C	KLB	9/20/17
Manually open the liquor line shutoff valve	The valve <u>is</u> open	KLB	9/20/17
Allow the digester to operate undisturbed for 1 hour	Start time: 12:30 End time: 1:30	KLB	9/20/17

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**3. Acceptance of Testing and Review**

Expected	Actual	Initials	Date
All actual results match the expected values.	All actual results <u>MATCH</u> the expected values.	KLB	9/20/17
The IQ section is complete with no deviations	The IQ section <u>IS</u> complete with no deviations	KLB	9/20/17
The OQ section is complete with no deviations	The OQ section <u>IS</u> complete with no deviations	KLB	9/20/17
The relevant standard working procedures are effective	The relevant standard working procedures <u>ARE</u> effective	KLB	9/20/17
List the procedure numbers	P12-PR-200-013 P17-PR-200-098 P17-PR-200-099 P12-PR-200-015 P12-PR-200-012 P12-PR-200-097	KLB	9/20/17
Results reviewed and accepted by		aw	9/20/17

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XIV. Protocol Deviation Report Log

Log each protocol deviation report in the table below. Attach PDRs to this protocol.

PDR #	Description	Protocol Section	Date Initiated	Date Resolved
			N/A	
			KLB	
			9/20/17	

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XV. Signature Identification Log

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

[illegible]

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