
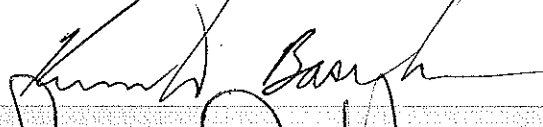


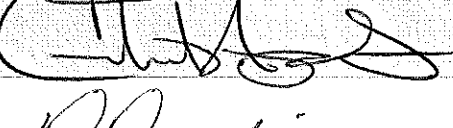



**GILES CHEMICAL ~ PREMIER MAGNESIA****Validation Protocol**

Title: Netzsch Filter Press Validation Final Report    Number: E17-VAL-PFR-220  
Owner: Kenneth Basehore    Revision: 0  
Effective Date: March 31, 2017    Page: 1 of 4

**I. Approvals**

Signing below indicates agreement that the execution of the Installation, Operational and Performance Qualification Protocol (E17-VAL-PIQ-560) for the Netzsch filter press, located at 102 Commerce Street, is complete and the process is validated.

Project Member	Functional Area	Signature	Date
Patrick Owen	Engineering		3/28/17
Kenneth Basehore	Engineering		3/28/17
Sammy Henson	Maintenance		3/28/17
Jason Bumgarner	Production		3-28-17
Matt Haynes	Operations		3-30-17
Deborah Durbin	Quality		3/31/17

A copy of the executed protocol will be attached to this report.

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# GILES CHEMICAL ~ PREMIER MAGNESIA

## Validation Protocol

Title: Netzsch Filter Press Validation Final Report      Number: E17-VAL-PFR-220

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## GILES CHEMICAL ~ PREMIER MAGNESIA

### Validation Protocol

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## II. Purpose

The purpose of this protocol is to certify with documented evidence that the Netzsch filter press (S/N 400-1322), 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 the Netzsch filter press, located at 102 Commerce Street at the Main Plant production facility.

## III. Summary

The Netzsch press filter was installed on site by contractors during late 2016, and finished during January of 2017. It is intended to receive a  $MgSO_4$  solution from the digesters. The solution is known as 'mud'. The mud is pumped through a series of filter plates at high pressure. The plates have filter cloths mounted on them, with a mesh tight enough to filter out insoluble impurities. The liquid flow from the filter press is pumped to storage tanks for use in the vacuum crystallizers. This liquid is called 'brine'.

The following tests were performed:

- The unit is installed correctly, and with access to all required points
- The squeeze pressure is appropriate
- The associated pumps are installed correctly, with the correct rotation
- The filter cloths do not have creases or folds
- Brine clarity matches current filter press production

All installation, operational and performance acceptance criteria were met as displayed in the attached executed protocol.

## IV. Conclusion

The results of the completed installation, operational and performance qualification protocol show that all acceptance criteria were met for all samples. All testing results provide documented evidence that the Netzsch press filter is installed, is operating and is performing as expected.

The tests were performed on 3/7/17 and 3/20/17, with training on the procedure updates completing on 3/17/17.

## V. Recommendations

It is recommended that the Netzsch press filter, located at the Giles Chemical Main Plant at 102 Commerce Street, Waynesville, NC 28786 be considered validated based on meeting the acceptance criteria of the IQ/OQ/PQ protocol.

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

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## VI. References

E17-VAL-PIQ-200: Netzsch Filter Press IQ/OQ/PQ Validation

P12-PR-200-020: Filter Press Set Points

P12-PR-200-018: Pressure Washing the Filter Presses

P12-PR-200-017: Operating the Filter Press

## VII. Summary of Brine Clarity Data

Six samples of brine were collected; a beginning, middle, end sample from two different presses (6 total), to determine two things:

- If the brine clarity changed throughout the course of a run within one filter press, and
- If the brine clarity changed between filter presses

To establish 'normal' operating condition, the Komline press was used (s/n AF-0228). The samples were pulled based on the following table:

Sample Number	Press	BME
1	Netzsch	Beginning
2	Netzsch	Middle
3	Netzsch	End
4	Komline	Beginning
5	Komline	Middle
6	Komline	End

Based on the laboratory testing, there is no difference between filter press runs, as well as within a single run. The brine clarity test passes.

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**GILES CHEMICAL ~ PREMIER MAGNESIA****Company Form**

Title: Document Approval

Number: Q12-PR-100-F002

Owner: Deborah Durbin

Revision: 4

Effective Date: 05/01/2016

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☒ **NEW DOCUMENT**   ☐ **REVISION**   ☐ **ARCHIVE**   ☐ **REVIEW (No Change)**☐ Human Resources☐ Quality Assurance☐ Manufacturing☐ IT☐ Customer Service☐ QA Laboratory☐ Repackaging☒ Engineering☐ Accounting☐ Safety☐ Maintenance☐ Other**Document Number** (new doc# assigned by QA): E17-VAL-PIQ-200**Revision #:** 0**Document Title:** Netzsch Filter Press Validation Protocol**Date Submitted:** 2/13/17**Owner:** Kenneth Basehore**Proposed Changes** (attach previous and revised documents or new document)

New document

**Reason for Changes** (be specific: new equipment, process change, 5 year review, general update, etc.)

New equipment

**Describe Type of Training Required if applicable** (email notification, routing of the document for reading and sign-off, classroom lecture, on the job, hands-on training, etc.)

None needed

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**GILES CHEMICAL ~ PREMIER MAGNESIA****Company Form**

Title: Document Approval

Number: Q12-PR-100-F002

Owner: Deborah Durbin

Revision: 4

Effective Date: 05/01/2016

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**Document Owner Signature:**

Printed Name and Title	Signature	Date
Kenneth Basehore - Process Engineer		2/13/17

**Department Approval Signatures:** Departments affected by new document or revisions

Printed Name and Title	Signature	Date
Patricia Owen Sec. Maint Mgr.		2/21/17
Sommy Joekerson Maint mgr.		2/21/17
Jason Bumgarner Plant Mgr.		2-21-17
C. Matt Haynes DR. OPER.		3-3-17

**Document Control Use Only:**☐ Training Records Received and Filed

Date: \_\_\_\_\_

☐ Document Approved

Effective Date: \_\_\_\_\_

Signature: \_\_\_\_\_

Revision Number: \_\_\_\_\_

Quality

Archival Date: \_\_\_\_\_

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

Title: Netzsch Filter Press Validation Protocol

Number: E17-VAL-PIQ-200

Owner: Kenneth Basehore

Revision: 0

Effective Date: 2/28/17

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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 Netzsch filter press (s/n 400-1322), located at 102 Commerce Street at the Main Plant production facility.

Project Member	Functional Area	Signature	Date
Patrick Owen	Engineering		2/24/17
Kenneth Basehore	Engineering		2/13/17
Sammy Henson	Maintenance		2/21/17
Jason Bumgarner	Production		2-21-17
Matt Haynes	Operations		3-3-17
Deborah Durbin	Quality		3/3/17

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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# GILES CHEMICAL ~ PREMIER MAGNESIA

## Validation Protocol

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## II. Purpose

The purpose of this protocol is to certify with documented evidence that Netzsch filter press model 1200 SP (s/n 400-1322) 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 the filter press, located at 102 Commerce Street at the Main Plant production facility.

## III. Background

The Netzsch filter press (s/n 400-1322) was installed on site by contractors during late 2016, and finished during January 2017. It is intended to receive a  $MgSO_4$  solution from the digesters. The solution is known as 'mud'. The mud is pumped through a series of filter screens pressed together with a hydraulic pump. The screens filter out insoluble impurities. The waste stream from the filter press is caked and hauled to a land fill. The product stream from the filter press (known as 'brine') is pumped to a storage tank, and is fed to the crystalizers to produce solid  $MgSO_4$  salt.

## 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:

- The unit is installed correctly, and with access to all required points
- The squeeze pressure is appropriate
- The associated pumps are installed correctly, with the correct rotation
- The filter cloths do not have creases or folds
- Brine clarity matches current filter press production

## V. System Description



1. The filter press is operated through a series of preprogrammed steps in a control interface.
2. The steps control the squeeze pressure, the product flow and flush timing.
3. Mud flows into the press, and brine is separated from insoluble impurities.

## VI. Scope

The IQ, OQ and PQ contained within this protocol is intended to certify with documented evidence that the Netzsch filter (s/n 400-1322) 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 the Netzsch filter (s/n 400-1322) is installed correctly.



#### b. Equipment and Materials

- Netzsch filter model 1200 SP (s/n 400-1322)
- Mud Feed Pump model PC196 (s/n 6357)
- Preheat pump model PC196 (s/n 4091)
- Squeeze pump model 84Z04053 (s/n 06F266W036G1)
- Cake wash pump model 3657 (s/n 5SS1M9E5)

#### c. Procedure

- Verify that the press is situated to allow sufficient room around the machine for access
- Verify that all pumps are situated to allow sufficient room for access
- Verify that the press is level

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- Verify that the pumps are level
- Verify that the electrical utilities fall within the manufacturers required ranges

d. Acceptance Criteria

Ensure that the installation is correct.

## 2. Operational Qualification (OQ)

a. Objective

The objective of the operational qualification is to ensure that the Netzsch press operates as intended by the manufacturer. 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

- Netzsch filter model 1200 SP (s/n 400-1322)
- Mud Feed Pump model PC196 (s/n 6357)
- Preheat pump model PC196 (s/n 4091)
- Squeeze pump model 84Z04053 (s/n 06F266W036G1)
- Cake wash pump model 3657 (s/n 5SS1M9E5)

c. Procedure

- Verify that the squeeze pressure is appropriate
- Verify that the associated pumps work properly
- Verify that the pumps have the correct rotation direction
- Verify that the cloths do not have creases or folds

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 the Netzsch press performs the functions required by Giles Chemical. This protocol will verify the following:

b. Equipment and Materials

- Netzsch filter model 1200 SP (s/n 400-1322)
- Mud Feed Pump model PC196 (s/n 6357)
- Preheat pump model PC196 (s/n 4091)
- Squeeze pump model 84Z04053 (s/n 06F266W036G1)
- Cake wash pump model 3657 (s/n 5SS1M9E5)

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c. Procedure

- Verify that the press proceeds through the programmed steps correctly
- Verify that the brine clarity is not different than existing press brine clarity

d. Acceptance Criteria

The PQ will be accepted if the brine clarity is not different than existing press brine clarity, and if the press proceeds through the programmed steps correctly.

## IX. Calibration

Verify that all instruments used are within the calibration dates.

- Calibrated multimeter

## X. References

None

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

Device	Calibration Date	Calibration Expiration	Verified By	Date
Multimeter Model: Fluke 114 S/N: 36250117WS	10/16	10/17	KLB	3/7/17

Expected	Actual	Pass/Fail	Verified By	Date
Netzsch Press Filter Model: 1200 SP S/N: 400-1322	NETZSCH PRESS FILTER MODEL: 1200 SP S/N: 400-1322	PASS	KLB	3/7/17
Mud Feed Pump Model: PC196 S/N: 6357	MUD FEED PUMP MODEL: PC196 S/N: 6357	PASS	KLB	3/7/17
Preheat Pump Model: PC196 S/N: 4091	PREHEAT PUMP MODEL: PC196 S/N: 4091	PASS	KLB	3/7/17
Squeeze Pump Model: 84Z04053 S/N: 06F266W036G1	SQUEEZE PUMP MODEL: 84Z04053 S/N: 06F266W036G1	PASS	KLB	3/7/17
Cake Wash Pump Model: 3657 S/N: 5SS1M9E5	CAKE WASH PUMP MODEL: 3657 S/N: 5SS1M9E5	PASS	KLB	3/7/17

**2. Acceptance Testing**

Expected	Actual	Pass/Fail	Verified By	Date
There is sufficient room around the press to allow access doors and panels to be opened	There <u>is</u> sufficient room around the press to allow access doors and panels to be opened.	PASS	KLB	3/7/17
There is sufficient room around the mud feed pump to allow maintenance	There <u>is</u> sufficient room around the mud feed pump to allow maintenance	PASS	KLB	3/7/17
There is sufficient room around the preheat pump to allow maintenance	There <u>is</u> sufficient room around the preheat pump to allow maintenance	PASS	KLB	3/7/17

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There is sufficient room around the squeeze pump to allow maintenance	There <u>15</u> sufficient room around the squeeze pump to allow maintenance	PASS	KLB	3/7/17
There is sufficient room around the cake wash pump to allow maintenance	There <u>15</u> sufficient room around the cake wash pump to allow maintenance	PASS	KLB	3/7/17
The press is level	The press <u>15</u> level	PASS	KLB	3/7/17
The mud feed pump is level	The mud feed pump <u>15</u> level	PASS	KLB	3/7/17
The preheat pump is level	The preheat pump <u>15</u> level	PASS	KLB	3/7/17
The squeeze pump is level	The squeeze pump <u>15</u> level	PASS	KLB	3/7/17
The cake wash pump is level	The cake wash pump <u>15</u> level	PASS	KLB	3/7/17
All four pumps are 230 VAC 3PH	All four pumps <u>ARE</u> 230 VAC 3PH	PASS	KLB	3/7/17

### 3. Acceptance of Testing and Review

Expected	Actual	Pass/Fail	Verified By	Date
All actual results match the expected values.	All actual results <u>MATCH</u> the expected values.	PASS	KLB	3/7/17
The relevant standard working procedures are approved	The relevant standard working procedures <u>ARE</u> approved	PASS	KLB	3/7/17
List the procedure numbers	P12-PR-200-017 P12-PR-200-018 P12-PR-200-020	PASS	KLB	3/7/17

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Results reviewed and accepted by

PASS

aw

3/22/17

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**XI. Operational Qualification (OQ)****1. Equipment**

Expected	Actual	Pass/Fail	Verified By	Date
Netzsch Press Filter Model: 1200 SP S/N: 400-1322	NETZSCH PRESS FILTER MODEL: 1200 SP S/N: 400-1322	PASS	KLB	3/7/17
Mud Feed Pump Model: PC196 S/N: 6357	MUD FEED PUMP MODEL: PC196 S/N: 6357	PASS	KLB	3/7/17
Preheat Pump Model: PC196 S/N: 4091	PREHEAT PUMP MODEL: PC196 S/N: 4091	PASS	KLB	3/7/17
Squeeze Pump Model: 84Z04053 S/N: 06F266W036G1	SQUEEZE PUMP MODEL: 84Z04053 S/N: 06F266W036G1	PASS	KLB	3/7/17
Cake Wash Pump Model: 3657 S/N: 5SS1M9E5	CAKE WASH PUMP MODEL: 3657 S/N: 5SS1M9E5	PASS	KLB	3/7/17

**2. Acceptance Testing**

Expected	Actual	Pass/Fail	Verified By	Date
The press is stopped	The press <u>IS</u> stopped	PASS	KLB	3/7/17
Press open filter	The press <u>OPENS</u> .	PASS	KLB	3/7/17
Ensure that none of the clothes have creases or folds	The cloths <u>DO NOT</u> have creases or folds	PASS	KLB	3/7/17
Press close filter	The press <u>CLOSES</u> .	PASS	KLB	3/7/17
From the Main Screen, press the 'Go to Advanced' button	The button <u>IS</u> pressed	PASS	KLB	3/7/17
The program steps are displayed	The program steps <u>ARE</u> displayed	PASS	KLB	3/7/17

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Record the program steps	PRE HEAT WASH LIQUOR PRODUCT FEED WATER FLUSH PRE SQUEEZE CAKE WASH FINAL SQUEEZE CORE BLOW SQUEEZE VENT	AIR BLOW AIR BLOW VENT CYCLE END	PASS	KLB	3/7/17
Navigate to the 'Filter Setpoints' screen	The 'Filter Setpoints' screen <u>is</u> displayed		PASS	KLB	3/7/17
Change the 'Feed Low Flow to End' setpoint to 50 GPM	The setpoint <u>is</u> changed		PASS	KLB	3/7/17
Change the 'Feed Low Flow to End' setpoint to 30 GPM	The setpoint <u>is</u> changed		PASS	KLB	3/7/17
Record the 'Final Squeeze Pressure'	<u>60</u> psi		PASS	KLB	3/7/17
Press start conveyor	The conveyor <u>STARTS</u> .		PASS	KLB	3/7/17
Press stop conveyor	The conveyor <u>STOPS</u> .		PASS	KLB	3/7/17

**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	3/7/17
The IQ section is complete with no deviations	The IQ section <u>is</u> complete with no deviations	KLB	3/7/17
Results reviewed and accepted by		aw	3/22/17

**Controlled Document**

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

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**XII. Performance Qualification (PQ)****1. Equipment**

Expected	Actual	Pass/Fail	Verified By	Date
<b>Netzsch Press Filter</b> Model: 1200 SP S/N: 400-1322	NETZSCH PRESS FILTER MODEL: 1200 SP S/N: 400-1322	PASS	KLB	3/20/17
<b>Mud Feed Pump</b> Model: PC196 S/N: 6357	MUD FEED PUMP MODEL: PC196 S/N: 6357	PASS	KLB	3/20/17
<b>Preheat Pump</b> Model: PC196 S/N: 4091	PREHEAT PUMP MODEL: PC196 S/N: 4091	PASS	KLB	3/20/17
<b>Squeeze Pump</b> Model: 84Z04053 S/N: 06F266W036G1	SQUEEZE PUMP MODEL: 84Z04053 S/N: 06F266W036G1	PASS	KLB	3/20/17
<b>Cake Wash Pump</b> Model: 3657 S/N: 5SS1M9E5	CAKE WASH PUMP MODEL: 3657 S/N: 5SS1M9E5	PASS	KLB	3/20/17

**2. Acceptance Testing**

Expected	Actual	Pass/Fail	Verified By	Date
<b>The press is stopped</b>	The press <u>IS</u> stopped	PASS	KLB	3/20/17
<b>Press open filter</b>	The press <u>OPENED</u> .	PASS	KLB	3/20/17
<b>Ensure that none of the clothes have creases or folds</b>	The cloths <u>DO NOT</u> have creases or folds	PASS	KLB	3/20/17
<b>Press close filter</b>	The press <u>CLOSED</u> .	PASS	KLB	3/20/17
<b>From the Main Screen, press the 'Go to Advanced' button</b>	The button <u>IS</u> pressed	PASS	KLB	3/20/17
<b>The program steps are displayed</b>	The program steps <u>ARE</u> displayed	PASS	KLB	3/20/17

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## GILES CHEMICAL ~ PREMIER MAGNESIA

## Validation Protocol

Title: Netzsch Filter Press Validation Protocol

Number: E17-VAL-PIQ-200

Owner: Kenneth Basehore

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Record the program steps	PRE HEAT WASH LIQUOR PRODUCT FEED WATER FLUSH PRE SQUEEZE CAKE WASH FINAL SQUEEZE CORE BLOW SQUEEZE VENT	AIR BLOW AIR BLOW VENT CYCLE END	PASS	KLB	3/20/17
Navigate to the 'Filter Setpoints' screen	The 'Filter Setpoints' screen <u>is</u> displayed		PASS	KLB	3/20/17
Record the 'Final Squeeze Pressure'	<u>60</u> psi		PASS	KLB	3/20/17
Press start conveyor	The conveyor <u>STARTS</u>		PASS	KLB	3/20/17
Press 'Start Filter'	The press <u>is</u> started		PASS	KLB	3/20/17
The press progresses through the program steps	The press <u>PROGRESSES</u> through the program steps		PASS	KLB	3/20/17
The mud feed pump has the correct rotation, and is working correctly	The mud feed pump <u>HAS</u> the correct rotation, and is working correctly		PASS	KLB	3/20/17
The preheat pump has the correct rotation, and is working correctly	The preheat pump <u>HAS</u> the correct rotation, and is working correctly		PASS	KLB	3/20/17
The squeeze pump has the correct rotation, and is working correctly	The squeeze pump <u>HAS</u> the correct rotation, and is working correctly		PASS	KLB	3/20/17
The cake wash pump has the correct rotation, and is working correctly	The cake wash pump <u>HAS</u> the correct rotation, and is working correctly		PASS	KLB	3/20/17
Collect a beginning brine sample for lab analysis	The sample <u>is</u> collected		PASS	KLB	3/20/17
Collect a middle brine sample for lab analysis	The sample <u>is</u> collected		PASS	KLB	3/20/17

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Collect an ending brine sample for lab analysis	The sample <u>15</u> collected	PASS	KLB	3/20/17
Collect a BME brine sample from a different filter press	The sample <u>15</u> collected Press s/n: <u>AF-0228</u>	PASS	KLB	3/20/17
The six samples show no difference in clarity	The six samples <u>SHOW NO</u> difference in clarity	PASS	KLB	3/20/17
The press is stopped	The press <u>HAS</u> stopped	PASS	KLB	3/20/17
Ensure that none of the clothes have creases or folds	The cloths <u>DO NOT</u> have creases or folds	PASS	KLB	3/20/17

## 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	3/20/17
The IQ section is complete with no deviations	The IQ section <u>15</u> complete with no deviations	KLB	3/20/17
The OQ section is complete with no deviations	The OQ section <u>15</u> complete with no deviations	KLB	3/20/17
The relevant standard working procedures are effective	The relevant standard working procedures <u>ARE</u> effective	KLB	3/20/17
List the procedure numbers	P12-PR-200-017 P12-PR-200-018 P12-PR-200-020	KLB	3/20/17

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Results reviewed and accepted by

aw

3/22/17

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### XIII. 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		
		3/22/17		

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**XIV. Signature Identification Log**

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

Name	Affiliation	Signature	Initials	Date
Kenneth Basehore	Engineering		KLB	3/7/17
Ashley Williams	Quality		aw	3/22/17

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