

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

Title: ACS Filter Press Validation Final Report

Number: E17-VAL-PFR-231

Owner: Kenneth Basehore

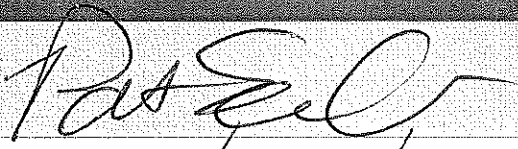

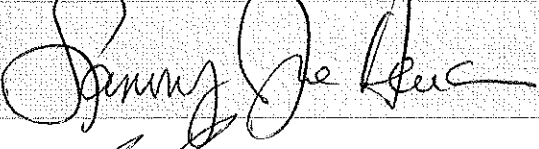
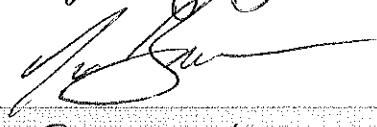


Revision: 0

Effective Date: June 9, 2017

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**I. Approvals**

Signing below indicates agreement that the execution of the Installation, Operational and Performance Qualification Protocol (E17-VAL-PIQ-230) for the ACS filter press, located at 102 Commerce Street, is complete and the process is validated.

| Project Member | Functional Area | Signature | Date |
|------------------|-----------------|--|---------|
| Patrick Owen | Engineering |  | 5/31/17 |
| Kenneth Basehore | Engineering |  | 5/31/17 |
| Sammy Henson | Maintenance |  | 5/31/17 |
| Jason Bumgarner | Production |  | 5-31-17 |
| Matt Haynes | Operations |  | 5/31/17 |
| Deborah Durbin | Quality |  | 5/31/17 |

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

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II. Purpose

The purpose of this protocol is to certify with documented evidence that the ACS filter press 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 ACS filter press, located at 102 Commerce Street at the Main Plant production facility.

III. Summary

The ACS press filter was installed on site by contractors during late 2007. 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 ACS press filter is installed, is operating and is performing as expected.

The tests were performed on 5/22/17 and 5/24/17.

V. Recommendations

It is recommended that the ACS 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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VI. References

E17-VAL-PIQ-230: ACS 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 | ACS | Beginning |
| 2 | ACS | Middle |
| 3 | ACS | End |
| 4 | Netzsch | Beginning |
| 5 | Netzsch | Middle |
| 6 | Netzsch | 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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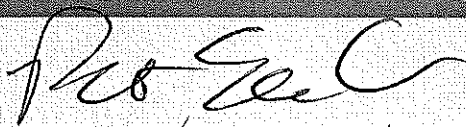
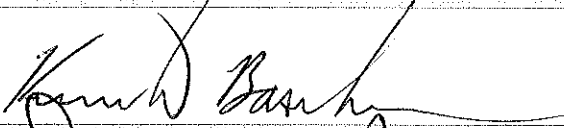


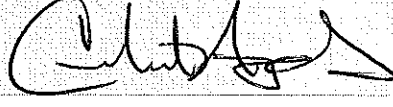

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**I. Approvals**

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

| Project Member | Functional Area | Signature | Date |
|------------------|-----------------|--|---------|
| Patrick Owen | Engineering |  | 5/15/17 |
| Kenneth Basehore | Engineering |  | 5/10/17 |
| Sammy Henson | Maintenance |  | 5/15/17 |
| Jason Bumgarner | Production |  | 5-15-17 |
| Matt Haynes | Operations |  | 5-15-17 |
| Deborah Durbin | Quality |  | 5-15-17 |

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 ACS filter press 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 ACS filter press was installed on site by contractors during late 2007. 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 crystallizers 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 ACS filter press 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.

VII. Roles and Responsibilities

1. Engineering
 - Write and issue the protocol

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- 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 ACS filter press is installed correctly.



b. Equipment and Materials

- ACS filter press
- Mud Feed Pump model MTX3196 (s/n 68401)
- Preheat pump model PC196 (s/n 6178)
- Squeeze pump model DVPF 6/6 B (s/n 290062362060GN)
- Cake wash pump model 3657 (J1402076)

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
- Verify that the pumps are 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.

2. Operational Qualification (OQ)

a. Objective

The objective of the operational qualification is to ensure that the ACS filter 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

- ACS filter press
- Mud Feed Pump model MTX3196 (s/n 68401)
- Preheat pump model PC196 (s/n 6178)
- Squeeze pump model DVPF 6/6 B (s/n 290062362060GN)
- Cake wash pump model 3657 (J1402076)

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



b. Equipment and Materials

- ACS filter press
- Mud Feed Pump model MTX3196 (s/n 68401)
- Preheat pump model PC196 (s/n 6178)
- Squeeze pump model DVPF 6/6 B (s/n 290062362060GN)
- Cake wash pump model 3657 (J1402076)

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

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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 | 10/2016 | 10/2017 | KLB | 5/22/17 |

| Expected | Actual | Pass/Fail | Verified By | Date |
|--|--|-----------|-------------|---------|
| ACS Press Filter | ACS PRESS FILTER | PASS | KLB | 5/22/17 |
| Mud Feed Pump Model: MTX3196 S/N: 68401 | MUD FEED PUMP MODEL: MTX3196 S/N: 68401 | PASS | KLB | 5/22/17 |
| Preheat Pump Model: PC196 S/N: 6178 | PREHEAT PUMP MODEL: PC196 S/N: 6178 | PASS | KLB | 5/22/17 |
| Squeeze Pump Model: DVPF 6/6 B S/N: 290062362060GN | SQUEEZE PUMP MODEL: DVPF 6/6 B S/N: 290062362060GN | PASS | KLB | 5/22/17 |
| Cake Wash Pump Model: 3657 S/N: J1402076 | CAKE WASH PUMP MODEL: 3657 S/N: J1402076 | PASS | KLB | 5/22/17 |

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 | 5/22/17 |
| 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 | 5/22/17 |
| 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 | 5/22/17 |
| There is sufficient room around the squeeze pump to allow maintenance | There <u>IS</u> sufficient room around the squeeze pump to allow maintenance | PASS | KLB | 5/22/17 |

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| 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 | 5/22/17 |
| The press is level | The press <u>15</u> level | PASS | KLB | 5/22/17 |
| The mud feed pump is level | The mud feed pump <u>15</u> level | PASS | KLB | 5/22/17 |
| The preheat pump is level | The preheat pump <u>15</u> level | PASS | KLB | 5/22/17 |
| The squeeze pump is level | The squeeze pump <u>15</u> level | PASS | KLB | 5/22/17 |
| The cake wash pump is level | The cake wash pump <u>15</u> level | PASS | KLB | 5/22/17 |
| All four pumps are 230 VAC 3PH | All four pumps <u>ARE</u> 230 VAC 3PH | PASS | KLB | 5/22/17 |

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 | 5/22/17 |
| The relevant standard working procedures are approved | The relevant standard working procedures <u>ARE</u> approved | PASS | KLB | 5/22/17 |
| List the procedure numbers | P12-PR-200-017 P12-PR-200-018 P12-PR-200-020 | PASS | KLB | 5/22/17 |
| Results reviewed and accepted by | | | AW | 5/22/17 |

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PREMIER
MAGNESIA, LLC**XI. Operational Qualification (OQ)****1. Equipment**

| Expected | Actual | Pass/Fail | Verified By | Date |
|--|--|-----------|-------------|---------|
| ACS Press Filter | ACS PRESS FILTER | PASS | KLB | 5/22/17 |
| Mud Feed Pump Model: MTX3196 S/N: 68401 | MUD FEED PUMP MODEL: MTX3196 S/N: 68401 | PASS | KLB | 5/22/17 |
| Preheat Pump Model: PC196 S/N: 6178 | PREHEAT PUMP MODEL: PC196 S/N: 6178 | PASS | KLB | 5/22/17 |
| Squeeze Pump Model: DVPF 6/6 B S/N: 290062362060GN | SQUEEZE PUMP MODEL: DVPF 6/6 B S/N: 290062362060GN | PASS | KLB | 5/22/17 |
| Cake Wash Pump Model: 3657 S/N: J1402076 | CAKE WASH PUMP MODEL: 3657 S/N: J1402076 | PASS | KLB | 5/22/17 |

2. Acceptance Testing

| Expected | Actual | Pass/Fail | Verified By | Date |
|---|--|-----------|-------------|---------|
| The press is stopped | The press <u>IS</u> stopped | PASS | KLB | 5/23/17 |
| Press open filter | The press <u>OPENED</u> . | PASS | KLB | 5/23/17 |
| Ensure that none of the clothes have creases or folds | The cloths <u>DO NOT</u> have creases or folds | PASS | KLB | 5/23/17 |
| Press close filter | The press <u>CLOSES</u> . | PASS | KLB | 5/23/17 |
| From the Main Screen, press the 'Go to Advanced' button | The button <u>IS</u> pressed | PASS | KLB | 5/23/17 |
| The program steps are displayed | The program steps <u>ARE</u> displayed | PASS | KLB | 5/23/17 |

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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 | 5/23/17 |
| Navigate to the 'Filter Setpoints' screen | The 'Filter Setpoints' screen <u>is</u> displayed | | PASS | KLB | 5/23/17 |
| Change the 'Feed Low Flow to End' setpoint to 50 GPM | The setpoint <u>is</u> changed | | PASS | KLB | 5/23/17 |
| Change the 'Feed Low Flow to End' setpoint to 30 GPM | The setpoint <u>is</u> changed | | PASS | KLB | 5/23/17 |
| Record the 'Final Squeeze Pressure' | <u>60</u> psi | | PASS | KLB | 5/23/17 |
| Press start conveyor | The conveyor <u>STARTS</u> . | | PASS | KLB | 5/23/17 |
| Press stop conveyor | The conveyor <u>STOPS</u> . | | PASS | KLB | 5/23/17 |

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 | 5/23/17 |
| The IQ section is complete with no deviations | The IQ section <u>is</u> complete with no deviations | KLB | 5/23/17 |
| Results reviewed and accepted by | | aw | 5/24/17 |

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

| Expected | Actual | Pass/Fail | Verified By | Date |
|--|--|-----------|-------------|---------|
| ACS Press Filter | ACS PRESS FILTER | PASS | KLB | 5/24/17 |
| Mud Feed Pump Model: MTX3196 S/N: 68401 | MUD FEED PUMP MODEL: MTX3196 S/N: 68401 | PASS | KLB | 5/24/17 |
| Preheat Pump Model: PC196 S/N: 6178 | PREHEAT PUMP MODEL: PC196 S/N: 6178 | PASS | KLB | 5/24/17 |
| Squeeze Pump Model: DVPF 6/6 B S/N: 290062362060GN | SQUEEZE PUMP MODEL: DVPF 6/6 B S/N: 290062362060GN | PASS | KLB | 5/24/17 |
| Cake Wash Pump Model: 3657 S/N: J1402076 | CAKE WASH PUMP MODEL: 3657 S/N: J1402076 | PASS | KLB | 5/24/17 |

2. Acceptance Testing

| Expected | Actual | Pass/Fail | Verified By | Date |
|---|--|-----------|-------------|---------|
| The press is stopped | The press <u>IS</u> stopped | PASS | KLB | 5/24/17 |
| Press open filter | The press <u>OPENS</u> . | PASS | KLB | 5/24/17 |
| Ensure that none of the clothes have creases or folds | The cloths <u>DO NOT</u> have creases or folds | PASS | KLB | 5/24/17 |
| Press close filter | The press <u>CLOSES</u> . | PASS | KLB | 5/24/17 |
| From the Main Screen, press the 'Go to Advanced' button | The button <u>IS</u> pressed | PASS | KLB | 5/24/17 |
| The program steps are displayed | The program steps <u>ARE</u> displayed | PASS | KLB | 5/24/17 |

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|---|---|--|------|-----|---------|
| Record the program steps | PRE HEAT WASH LIQUOR PRODUCT FEED WATER FLUSH PRESQUEEZE CAKE WASH FINAL SQUEEZE CORE BLOW SQUEEZE VENT | AIR BLOW AIR BLOW VENT CYCLE END | PASS | KLB | 5/24/17 |
| Navigate to the 'Filter Setpoints' screen | The 'Filter Setpoints' screen <u>IS</u> displayed | | PASS | KLB | 5/24/17 |
| Record the 'Final Squeeze Pressure' | <u>60</u> psi | | PASS | KLB | 5/24/17 |
| Press start conveyor | The conveyor <u>STARTS</u> | | PASS | KLB | 5/24/17 |
| Press 'Start Filter' | The press <u>IS</u> started | | PASS | KLB | 5/24/17 |
| The press progresses through the program steps | The press <u>PROGRESSES</u> through the program steps | | PASS | KLB | 5/24/17 |
| 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 | 5/24/17 |
| 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 | 5/24/17 |
| 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 | 5/24/17 |
| 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 | 5/24/17 |
| Collect a beginning brine sample for lab analysis | The sample <u>IS</u> collected | | PASS | KLB | 5/24/17 |
| Collect a middle brine sample for lab analysis | The sample <u>IS</u> collected | | PASS | KLB | 5/24/17 |

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

Title: ACS Filter Press Validation Protocol

Number: E17-VAL-PIQ-230

Owner: Kenneth Basehore

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| | | | | |
|--|--|------|-----|---------|
| Collect an ending brine sample for lab analysis | The sample <u>is</u> collected | PASS | KLB | 5/24/17 |
| Collect a BME brine sample from a different filter press | The sample <u>is</u> collected Press s/n: <u>400-1322</u> | PASS | KLB | 5/24/17 |
| The six samples show no difference in clarity | The six samples <u>SHOW NO</u> difference in clarity | PASS | KLB | 5/24/17 |
| The press is stopped | The press <u>is</u> stopped | PASS | KLB | 5/24/17 |
| Ensure that none of the clothes have creases or folds | The cloths <u>DO NOT</u> have creases or folds | PASS | KLB | 5/24/17 |

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 | 5/24/17 |
| The IQ section is complete with no deviations | The IQ section <u>is</u> complete with no deviations | KLB | 5/24/17 |
| The OQ section is complete with no deviations | The OQ section <u>is</u> complete with no deviations | KLB | 5/24/17 |
| The relevant standard working procedures are effective | The relevant standard working procedures <u>ARE</u> effective | KLB | 5/24/17 |
| List the procedure numbers | P12-PR-200-017 P12-PR-200-018 P12-PR-200-020 | KLB | 5/24/17 |

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Results reviewed and accepted by

QW

5/26/17

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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 | |
| | | | 5/24/17 | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |

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