

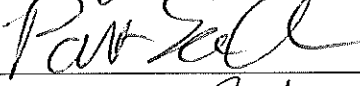
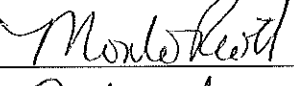
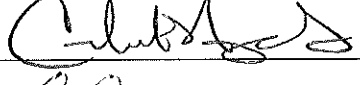
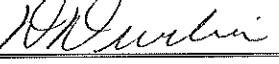
	GILES CHEMICAL ~ PREMIER MAGNESIA		
	Validation Protocol		
	Title: Repackaging Scented Salt Cleaning Procedure Validation Protocol	Number: E13-VAL-RCV-751	
	Owner: Bryan Elchert	Revision: 0	
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## Approvals

Signing below indicates agreement that the protocol is ready for execution of the Scented Salt Cleaning Procedure Validation for Giles Repackaging located at 396 Smathers Street, Waynesville, NC.

Project Team Member	Functional Area	Signature	Date
Bryan Elchert	QA Laboratory		7/15/13
Patrick Owen	Engineering		7/15/13
Monte Plott	Production		7/15/13
Matt Haynes	Operations		7/15/13
Deborah Durbin	Quality		7/15/13

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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**GILES CHEMICAL ~ PREMIER MAGNESIA****Validation Protocol****Title:** Repackaging Scented Salt Cleaning  
Procedure Validation Protocol**Number:** E13-VAL-RCV-751**Owner:** Bryan Elchert**Revision:** 0**Effective Date:** June 25, 2013**Page:** 2 of 10**PREMIER**  
**MAGNESIA, LLC**

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## I. PURPOSE:

The purpose of this protocol is to certify with documented evidence that the cleaning procedure for Scented Salt (Repackaging Scented Salt Cleaning Procedure, R12-PR-100-034), when followed properly, sufficiently removes all fragrance oil and cleaning residues to an acceptable level.

## II. BACKGROUND:

Scented Epsom Salt products are produced at the Repackaging Facility. The scent is injected into the pouches at the point of filling with a special funnel and nozzle. Until the salt reaches that point, it is suitable for human consumption. A product changeover is performed any time one of these lines is changed from scented product to a different scented product or scented product to product suitable for human consumption. Dawn Dishwashing Liquid is used for cleaning followed by an Isopropyl Alcohol mist. This study will evaluate the amount of Fragrance Oil and Dawn Dishwashing Liquid residue left after cleaning.

## III. SCOPE

This validation covers the Scented Salt Cleaning Procedure for Repackaging.

## IV. ROLES AND RESPONSIBILITIES

### 1. QA Laboratory

- ❖ Write and issue the protocol.
- ❖ Investigate protocol deviation reports.
- ❖ Test the rinse sample provided.
- ❖ 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.
- ❖ Source and provide clean sample bottles.
- ❖ Execute the Cleaning Procedure correctly.
- ❖ Gather and properly label rinse samples.
- ❖ Review, approve, and store the final report.

### 3. Production & Quality Assurance

- ❖ Review and approve the protocol.
- ❖ Review and approve the final report.

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## V. TEST PROGRAM

### A. AT REPACKAGING

**Objective:** Perform the standard Repackaging Scented Salt Cleaning Procedure 16 times to account for variability in the cleaning process. After complete cleaning, rinse the funnel with distilled water, and evaluate cleaning effectiveness.

#### Equipment/Materials

Stainless Steel Funnel Attachment

Repackaging Scented Salt Cleaning Procedure (and associated materials) R12-PR-100-034

Distilled Water

Clean 250 mL Graduated Cylinder

Clean Disposable Funnels

Sample Bottles



#### Procedure

1. After the Stainless Steel Funnel was exposed to fragranced salt, the funnel was washed using 3ml Dawn Dishwashing Liquid per 10gal of water. The funnel was washed for 2.5 minutes in the Dawn solution with the temperature between 37-46°C.
2. The funnel was then rinsed with tap water for 1.5 minutes.
3. After rinsing, the funnel was dried using a cotton cloth followed by wiping with a lint free paper towel to remove any lint debris.
4. The funnel was then rinsed with 100 mL of Deionized water that was collected in a clean beaker.
5. The 100mL of water collected was then poured into a sample bottle and labeled.
6. For a Reagent Blank, 100mL of distilled water was collected in the beaker and poured into a sample bottle and labeled.

### B. AT QUALITY ASSURANCE LAB

1. Prepare a 10 ppm Standard of Known Concentration.
2. Analyze samples in triplicate on the Hach DR5000 Spectrophotometer using Hach Method 10129 for Low Range (0.3-20ppm) Total Organic Carbon (TOC).
3. Report the results.

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## VI. ACCEPTANCE CRITERIA

The cleaning procedure will be considered validated if the following criteria are all met:

1. The test result for the Blank Sample is 0 ppm.
2. The test result for the individual Samples is 1.0 ppm TOC or less.
3. The test result for the 10 ppm TOC Standard of Known Concentration has a value of 9.1-10.9 ppm for a 95% Confidence Limits of Distribution.

## VII. CALIBRATION

Verify that all instrumentation that requires calibration is calibrated.

- Hach DR5000 Spectrophotometer

## VIII. REFERENCE:

*Repackaging Scented Salt Cleaning Procedure (R12-PR-100-0XX)*

*Hach Method 10129, "Total Organic Carbon", Hach Company, Loveland, Colorado*

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**ATTACHMENT I.      Repack Scented Salt Cleaning Validation Data Sheet**

[illegible]

Reviewed By:

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

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**ATTACHMENT II. CALIBRATION VERIFICATION**

Equipment	Serial #	Calibration Date	Calibration Due Date	Verified By	Date
Hach Spectrophotometer					

Reviewed By: \_\_\_\_\_

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

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### ATTACHMENT III - PROTOCOL DEVIATION REPORT LOG



Log each Protocol Deviation Report in the table below. Attach the PDRs to this Attachment.

[illegible]

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

**General Information**

System Name: \_\_\_\_\_ Protocol Number: \_\_\_\_\_  
 Deviation Report Number: \_\_\_\_\_ Protocol Step & Page No.: \_\_\_\_\_

**Instructions**

InstructionsThe 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.

1. Reference the relevant protocol number, step and page number of the noted deviation above.
2. Complete the below listed sections. If necessary, use additional pages and attach any supporting info.
3. Include the original PDR(s) with the protocol as an attachment. Summarize the impact of the deviation in the Validation Report.

---

Description of Deviation:

Investigation Evaluation and Results:

Corrective Action and Resolution:

---

Overall Investigation Review:

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

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### ATTACHMENT V. SIGNATURE IDENTIFICATION LOG SHEET

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

Name	Affiliation	Signature	Initial	Date

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