Approvals

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

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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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.

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.

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

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

TOC ANALYSIS (ppm)				
Sample	**Scan #1**	**Scan #2**	**Scan #3**	**Pass/Fail**
Blank	0.0	0.0	0.0	Pass
Reagent Blank	0.2	0.2	0.2	Pass
10 ppm Standard	9.3	9.3	9.4	Pass
Sample #1	0.1	0.1	0.1	Pass
Sample #2	0.5	0.6	0.6	Pass
Sample #3	1.0	1.0	1.0	Pass
Sample #4	0.6	0.6	0.6	Pass
Sample #5	0.0	0.1	0.0	Pass
Sample #6	0.2	0.2	0.2	Pass
Sample #7	0.9	1.0	1.0	Pass
Sample #8	0.7	0.7	0.7	Pass
Sample #9	0.6	0.6	0.6	Pass
Sample #10	0.1	0.2	0.2	Pass
Sample #11	0.0	0.0	0.0	Pass
Sample #12	0.2	0.2	0.2	Pass
Sample #13	0.5	0.5	0.5	Pass
Sample #14	0.0	0.0	0.0	Pass
Sample #15	0.0	0.0	0.0	Pass
Sample #16	0.2	0.2	0.2	Pass
Comments:				

DISCUSSION:

The blank sample concentration was measured to be 0 ppm and all of the samples tested were found to have TOC concentrations of 1.0 ppm or less. In addition, the 10 ppm Standard of Known Concentration was found to be 9.3 ppm. This falls within the 9.1-10.9 range for a 95% Confidence Limits of Distribution. The collected data meets all acceptance criteria and therefore validates the Repackaging Scented Salt Cleaning Procedure as described.

Reviewed By: _____ **Date:** 7/15/2013

ATTACHMENT II. CALIBRATION VERIFICATION

Equipment	Serial #	Calibration Date	Calibration Due Date	Verified By	Date
Hach Spectrophotometer	1396156	7/12/13	7/14	BE	7/15/13

Reviewed By: _____ **Date:** 7/15/2013

ATTACHMENT III - PROTOCOL DEVIATION REPORT LOG

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

PDR #	DESCRIPTION	DATE INITIATED	DATE RESOLVED
Comments:			

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: _____

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
Bryan Elchert	Giles Chemical		BE	7/15/13
Lee Cagle	cGMP Coordinator		LC	7/15/2013