Approvals

Signing below indicates agreement that the protocol is ready for execution of the Swab Test 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		7/26/13
Patrick Owen	Engineering		7/26/13
Monte Plott	Production		7/26/13
Matt Haynes	Operations		7/26/13
Deborah Durbin	Quality		7/26/13

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 Swab Test procedure for Swabbing After Scented Salt (Repackaging Swab Test Procedure, R12-PR-100-015), when followed properly, confirms that all fragrance oil has been removed or decreased to an acceptable level. In this procedure, Thin Layer Chromatography (TLC) is used to confirm that all fragrance oil is removed after cleaning. The TLC results will be confirmed using Total Organic Carbon (TOC) analysis and the procedure thereby validated.

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. After cleaning, the lines are swabbed and Thin Layer Chromatography (TLC) is performed to confirm the removal of fragrance oil. Total Organic Carbon (TOC) analysis is then used to confirm and validate the results of the TLC analysis.

III. SCOPE

This validation covers the Swabbing After Scented Salt Testing Procedure. The result of the Swab Test, which utilizes Thin Layer Chromatography (TLC), is further validated using Total Organic Carbon (TOC) analysis.

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: After the stainless steel funnel attachment was cleaned and swabbed, the samples were ready for analysis. In this case, 16 samples were analyzed by TLC to verify the removal of fragrance oil. The TLC results are then confirmed by TOC analysis.

Equipment/Materials

Repackaging Swab Test Cleaning Procedure (and associated materials) R12-PR-100-015

Swabs

Isopropyl Alcohol

TLC Silica Plate

Development Chamber

Black Light

Procedure

1. Using a pencil, a line is drawn about 1 inch from the bottom of the TLC Plate. A sample number is written below the line. The sample is then 'spotted' on the line above the corresponding sample number.
2. A sample of fragrance oil is spotted on the line to be used as a control.
3. After the fragrance oil (control sample), and the samples were 'spotted' on the TLC plate, the plate is put into the development chamber containing Isopropyl Alcohol and covered. The Isopropyl Alcohol is then allowed to migrate up the TLC plate.
4. After the Isopropyl Alcohol has migrated up the plate for a distance of about 4 inches, remove the TLC plate from the development chamber and allow it to dry.
5. When the TLC plate is dry, the lights are shut off and the black light is turned on. While holding the black light over the TLC plate, look for spots on the plate that fluoresce. About 2 inches above the place where the fragrance oil was 'spotted' will be a spot that fluoresces. This is the fragrance oil that migrated with the Isopropyl Alcohol up the TLC Plate.
6. Examine the TLC plate where the samples were 'spotted' for evidence of fluorescence. If there is no evidence of fluorescence in these areas, then no fragrance oil is present in the sample.
7. If there is evidence of fragrance oil in a sample, repeat this procedure using the suspect sample and fragrance oil as a control.

B. AT QUALITY ASSURANCE LAB

1. Prepare a 10 ppm Standard of Known Concentration.
2. Analyze TLC samples in triplicate on the Hach DR5000 Spectrophotometer using Hach Method 10129 for Low Range (0.3-20ppm) Total Organic Carbon (TOC).
3. Compare the TOC results to the TLC results. The results of both tests should confirm that there is no fragrance oil present.
4. Report the results.

VI. ACCEPTANCE CRITERIA

The Swab Test TLC results will be considered validated if the following criteria are all met:

1. The test result for the TOC Blank Sample is 0 ppm.
2. The test result for the individual TLC 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. **TLC Results Validation Data Sheet**

Sample	Is there Fragrance Oil present? (Y/N)	Pass/Fail
Fragrance Oil (Control)	No	Pass
Sample #1	No	Pass
Sample #2	No	Pass
Sample #3	No	Pass
Sample #4	No	Pass
Sample #5	No	Pass
Sample #6	No	Pass
Sample #7	No	Pass
Sample #8	No	Pass
Sample #9	No	Pass
Sample #10	No	Pass
Sample #11	No	Pass
Sample #12	No	Pass
Sample #13	No	Pass
Sample #14	No	Pass
Sample #15	No	Pass
Sample #16	No	Pass
Comments:	All samples tested negative for the presence of Fragrance Oil.	

Reviewed By: _____ **Date:** 7/26/2013

ATTACHMENT II. TOC Results 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 TLC test results indicated that all the samples were negative for the presence of fragrance oil. The TLC results were confirmed by the TOC test results. The blank sample concentration was measured to be 0 ppm TOC and all of the TLC samples tested were found to have TOC concentrations of 1.0 ppm or less. In addition, the 10 ppm TOC 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 Swab Test Procedure as described.

Reviewed By: _____ **Date:** 7/26/2013

ATTACHMENT III. CALIBRATION VERIFICATION

Equipment	Serial #	Calibration Date	Calibration Due Date	Verified By	Date
Hach Spectrophotometer	1396156	7/12/13	7/14	BE	7/26/13

Reviewed By: _____ Date: 7/26/2013

ATTACHMENT IV - 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 V. 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 VI. 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	By EL	BE	7/26/13
Lee Cagle	cGMP Coordinator	Lee Cagle	LC	7/26/13