Approvals:

Signing below indicates agreement that the protocol is ready for execution of the Swab Test Mobile Phase Validation for Giles Repackaging located at 396 Smathers Street, Waynesville, NC.

Project Team Member	Functional Area	Signature	Date
Hunter Douglas	QA Laboratory		03/23/2015
Patrick Owen	Engineering		3/23/15
Matt Haynes	Operations		3/23/15
Deborah Durbin	Quality		3/23/15

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 changing of the mobile phase from Isopropyl Alcohol to Cyclohexane in the Swab Test procedure for *Residual Fragrance Test using TLC (R12-PR-100-015)*, when followed properly, provides better separation and resolution than the original method. The two mobile phases will be compared side by side with a series of blank and spiked samples, if both reagents show the same results the substitution of mobile phases will be 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 from scented product to product suitable for human consumption. Dawn dishwashing liquid is used for cleaning followed by an IPA mist. After cleaning, the lines are swabbed and Thin Layer Chromatography (TLC) is performed to confirm the removal of fragrance oil.

III. SCOPE:

This validation covers the changing of mobile phase from IPA to Cyclohexane in the *Residual Fragrance Test using TLC (R12-PR-100-015)* procedure. The results of the swab test using both mobile phases will be compared side-by-side which will thereby validate the change.

IV. ROLES AND RESPONSIBILITIES:

1. QA Laboratory
   - Write and issue the protocol
   - Investigate protocol deviations reports
   - Prepare standards and blanks
   - Perform testing
   - Review and compile raw data
   - Write and distribute final report for approval
2. Quality Assurance
   - Review and approve the protocol
   - Review and approve raw data and notifications
   - Review, approve, and retain the final report
3. Production & Quality Assurance
   - Review and approve the protocol
   - Review and approve the final report

V. TEST PROGRAM:

A. QA Laboratory

Objective: A blank of IPA and three 10ppm scented oil standards in IPA will be made and will be analyzed by TLC in both the original IPA mobile phase and the Cyclohexane mobile phase separately. 30 spots will be placed on each plate, 9 of each of the spiked standards and 3 of the blank. The results are to be compared side-by-side for conformity.

Equipment/Materials:
Residual Fragrance Test using TLC (R12-PR-100-015)
Spiked Standards
Blank Standard
Isopropyl Alcohol
Cyclohexane
(2) TLC Silica Gel Plates
Developing Chamber
Black Light
Micropipettes
Ruler
Pencil

Procedure:

1. Prepare the spiked standards by diluting 150uL of each scented oil to 15mL with IPA.
2. Select a TLC plate that is clean and un-marred by scratches or chips. Then, using a pencil and a ruler draw a strait, faint line (the spotting line) 1cm from the bottom of the plate.
3. Score the plate 10cm from the bottom of the plate so the mobile phase does not overshoot the desired distance.
4. Make 30 hash marks along the spotting line that are equidistance from one another.
5. Spot the TLC plate where the spotting line and the hash marks intersect with 10uL of the 10ppm standard and the blank. *(Note: There should only be one spot per hash mark. Each standard should be spotted 9 times and the blank should be spotted 3 times each.)*
6. Add >1cm of the IPA mobile phase to the TLC chamber and place the plate gently into the chamber spotted side down. *(Note: Ensure the spots are completely dry before putting the plate in the developing chamber.)*
7. Allow mobile phase to travel to the score mark and remove the plate.
8. Allow plate to dry completely and expose the plate to the black light. Mark the spots that are seen with the pencil and reserve the plate for comparison and storage.
9. Repeat steps 2-8 using Cyclohexane as mobile phase instead of IPA.

10. Compare the results from the two runs. Report results.

VI. ACCEPTANCE CRITERIA

The change of mobile phase will be considered validated if the following criteria are all met:

1. The blank spots show as negative
2. The 10ppm spiked standard spots show as positive
3. The test results for the new mobile phase mirror the results of the old mobile phase.

VII. REFERENCE

Residual Fragrance Test using TLC (R12-PR-100-015)

ATTACHMENT I – TLC RESULTS VALIDATION DATA SHEET

Mobile Phase Results					
Sample	**Type**	**Result (IPA)**	**Result (Cyclohexane)**	**Initial / Date**	**Reviewed / Date**
Sample #1	Blank				
Sample #2	Blank				
Sample #3	Blank				
Sample #4	Mane Eucalyptus Std				
Sample #5	Mane Eucalyptus Std				
Sample #6	Mane Eucalyptus Std				
Sample #7	Mane Eucalyptus Std				
Sample #8	Mane Eucalyptus Std				
Sample #9	Mane Eucalyptus Std				
Sample #10	Mane Eucalyptus Std				
Sample #11	Mane Eucalyptus Std				
Sample #12	Mane Eucalyptus Std				
Sample #13	Mane Lavender Std				
Sample #14	Mane Lavender Std				
Sample #15	Mane Lavender Std				
Sample #16	Mane Lavender Std				
Sample #17	Mane Lavender Std				
Sample #18	Mane Lavender Std				
Sample #19	Mane Lavender Std				
Sample #20	Mane Lavender Std				
Sample #21	Mane Lavender Std				
Sample #22	Arylessence Soothing Lavender				
Sample #23	Arylessence Soothing Lavender				
Sample #24	Arylessence Soothing Lavender				
Sample #25	Arylessence Soothing Lavender				
Sample #26	Arylessence Soothing Lavender				
Sample #27	Arylessence Soothing Lavender				
Sample #28	Arylessence Soothing Lavender				
Sample #29	Arylessence Soothing Lavender				
Sample #30	Arylessence Soothing Lavender				

ATTACHMENT II – PROTOCOL DEVIATIONS REPORT LOG

Log each Protocol Deviation Report in the table below. Attach the PDR's to this Attachment.

PDR #	DESCRIPTION	DATE INITIATED	DATE RESOLVED
Comments:			

ATTACHMENT III – PROTOCOL DEVIATION REPORT (PDR)

General Information

System Name: _____ Protocol Number: _____

Deviation Report Number: _____ Protocol Step & Page No.: _____

Instructions

The validation specialist assigns a sequential report number for each deviation with a specific protocol. For example, 001, 002, ect. 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 the 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.

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Description of Deviation:

Investigation Evaluation and Results:

Corrective Action and Resolution:

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Overall Investigation Review:

Prepared By: _____ Date: _____

ATTACHMENT IV – SIGNATURE IDENTIFICATION LOG SHEET

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

Name	Affiliation	Signature	Initial	Date
Hunter Douglas	QA Laboratory		HD	03/23/15
Ashley Williams	QA Laboratory	Ashley Williams	AW	3/23/15