Standard Operating Procedure for Sample Preparation of Non-aerosol Consumer Products and the Non-propellant Portion of Aerosol Consumer Products

SAS14
Revision 0.1

Northern Laboratory Branch
Monitoring and Laboratory Division

Approval Signatures	Approval Date
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Table of Contents

Standard Operating Procedure for Sample Preparation of Non-aerosol Consumer Products and the Non-propellant Portion of Aerosol Consumer Products

1 Introduction

This procedure describes how to prepare a non-aerosol sample or the non-propellant portion of an aerosol sample for analysis under Method 310 as required by the California Consumer Products Regulations. SAS13 describes the overall process by which Method 310 is performed, including the requirement that aerosol samples must first follow procedures outlined in SAS05 for propellant collection and can weights prior to aliquoting the non-propellant portion. Following the preparation of non-aerosol samples and non-propellant portion of aerosol samples by this procedure, the sample aliquots and sample dilutions may be analyzed as described in SAS13 in the following Standard Operating Procedures (SOP): SAS01, SAS02, SAS03, SAS04, SAS06, SAS07, SAS09, SAS15.

2 Summary of Method

This procedure describes the aliquoting of non-aerosol samples or the non-propellant portion of aerosol samples into archive and sample aliquot vials. This procedure further outlines preparing sample dilutions from the aliquot vial contents that are required for analysis by SAS02, SAS03, SAS04, SAS07, SAS09 and SAS15. A laboratory sample of known concentration, the Batch Sample (BS), is also prepared for analysis.

3 Acronyms and Definitions

Acronym or Term	Definition
ACS Grade	Chemicals meeting standards set by the American Chemical Society.
aliquot	A representative portion of a non-aerosol sample or the non-propellant portion of an aerosol sample.
analytical batch	A set of samples analyzed together as a group for a particular analysis.
archive aliquot	An aliquot of the sample retained per the sample retention policy for archival and re-test purposes.
ASTM	American Standards for Testing and Materials
Batch Sample (BS)	A laboratory prepared sample aliquot of known concentration for QC evaluation under Method 310.
CARB	California Air Resources Board
duplicate	A second analysis of a sample submitted for analysis under Method 310.

Acronym or Term	Definition
duplicate aliquot	An additional sample aliquot from the same sample carried through all steps of the sampling and analytical procedures of Method 310 in an identical manner.
ID	Identification
LIMS	Laboratory Information Management System
LIMS Manual	Consumer Products Database Special Analysis Section (Oracle Database and Applications Manual for LIMS)
MPA	1-methoxy-2-propanol
NLB	Northern Laboratory Branch
QC	Quality Control
QCM	Quality Control Manual
Rpm	Rotations per minute
sample	The sample submitted for analysis under Method 310.
sample aliquot	The sample aliquot is any aliquot used for analysis, and includes the duplicate aliquot, the Batch Sample, or any archive aliquot undergoing a re-test.
sample batch	A set of samples analyzed together under Method 310.
sample dilution	Dilution made from the sample aliquot.
sd	Standard deviation
SOP	Standard Operating Procedure
VOC	Volatile Organic Compound(s)

4 Interferences

4.1 Consumer product packaging may prevent separation of the product from the delivery system. In these cases, only qualitative data will be included on the report per SAS13.

4.2 Samples may not dissolve in solution. In cases where a sample dilution cannot be prepared and analyses requiring dilution cannot be performed, record this in LIMS and on the report to the client.

4.3 Samples may be non-homogenous and not yield a representative aliquot. In these cases, record this in LIMS and on the report to the client.

4.4 Highly volatile samples can evaporate quickly. To minimize this effect, limit the amount of time the vials and containers are open. Additionally, the analyst may add approximately 1-5mL of the solvent being used in the dilution to the volumetric flask, then tare the volumetric flask prior to sample introduction.

4.5 Entrained propellant can present challenges in obtaining a sample dilution weight as it off-gasses. Limit the amount of time the vials and containers are open.

4.6 Shave gels are designed to foam once dispensed and must be kept under refrigeration to minimize this effect.

5 Personnel Qualifications

5.1 Prior to performing this method, new personnel must be trained by staff with detailed knowledge of this method. Personnel must be trained to understand the program's requirements per any applicable State and federal regulations and/or guidance, and this SOP. Personnel will also be trained on how to safely and properly operate the equipment needed to perform the method, the quality assurance components, and LIMS functionality pertaining to the program.

5.2 Personnel should provide an initial demonstration of capability prior to performing this method on real-world samples (i.e., data for record).

5.3 Training will be documented and maintained by the laboratory supervisor.

6 Safety Requirements

6.1 All personnel must follow the general health and safety requirements found in NLB's Chemical Hygiene Plan.

6.2 Analysts should acknowledge any sample labeling for safety warnings and take appropriate safety measures.

6.3 Ensure engineering controls are in place and operating (i.e., fume hood or fume extraction arm).

6.4 The walk-in refrigerator is equipped with a safety release on the door to prevent locking persons inside. Personnel should wear appropriate personal protective equipment (PPE) which may include but not limited to clothing for a low temperature environment, non-slip footwear, and hearing protection.

7 Hazardous Waste

7.1 For any sample containers, evaluate the sample contents for guidance on proper disposal management. Samples should be segregated/disposed of by chemical category. Waste samples should be categorized by halogenated or non-halogenated organic solvents, acidic aqueous, caustic aqueous, or in some cases, the product may contain a chemical requiring special handling or disposal. If a product characterization is uncertain, consult with the NLB Health and Safety Coordinator (H&SC) or the Industrial Hygiene Safety Section.

Satellite carboys used for temporary storage should not be allowed to reach more than 75% capacity before either moving to the main hazardous waste storage area or transfer to the appropriate bulk storage drum. The NLB H&SC should be notified when any satellite container is moved to the main storage area and when waste is ready for pickup by the hazardous waste disposal contractor.

Store empty sample containers and packaging in a secure location until release of custody to the client.

7.2 Archive aliquots should be segregated/disposed of by chemical category as described in Section 7.1 for samples.

7.3 Contents of sample aliquot vials are to be disposed of in the same manner as excess sample once analysis under Method 310 is complete.

7.4 Sample dilutions are to be disposed of in the consumer products waste container in the satellite hazardous waste accumulation area once analysis under Method 310 is complete.

8 Equipment and Supplies

8.1 40mL Amber Vials, screw top with caps

8.2 20mL Vials, screw top with caps

8.3 8mL Vials, screw top with caps

8.4 2mL Autosampler Vials with caps

8.5 16mL Vials, screw top with caps

8.6 Autosampler vial cap crimper

8.7 Autosampler vial cap decrimper

8.8 Vial racks, various sizes

8.9 Volumetric Flasks, 1mL, 5mL, 10mL, and 500mL

8.10 Analytical Balance, capacity of at least 200g x 0.00001g readability (e.g., Mettler XP205)

8.11 Laboratory Work Station for analytical balance

8.12 Software for data transfer and collection (e.g., BalanceTalk, Excel, LabX)

8.13 Top-Loader Balance, capacity of at least 1000g x 0.001g readability

8.14 1.0g Mass, ASTM class 1 or better

8.15 Laboratory Information Management System (LIMS)

8.16 Laboratory vented bench top enclosure

8.17 Pipettors, 1000µL and 5000µL with tips

8.18 Transfer Tubes, disposable, 3-5mL capacity

8.19 Transfer Pipettes, disposable

8.20 Pasteur Pipettes, disposable with bulbs

8.21 Stirring rods

8.22 Scoopulas/spatulas

8.23 Syringes, disposable, 3mL, 20mL, and 60mL

8.24 Pliers

8.25 Can openers

8.26 Screwdrivers

8.27 Cutting tools, various (e.g., snips, scissors, etc.)

8.28 Forceps

8.29 Pipe cutters

8.30 Beakers and Tri-Pours

8.31 Task wipes (e.g., Kimwipes)

8.32 Vortex mixer (e.g., Vortex Genie 2)

8.33 Homogenizer (e.g., IKA T8.01 S1)

8.34 Sonicator (e.g., Bransonic Ultrasonic Cleaner 2510R-MT)

8.35 Syringe Filter (e.g., 25mm GD/X Disposable Filters, Glass Microfiber GMF with Polypropylene Housing, pore size 0.45µL)

8.36 Gloves, non-powdered nitrile or suitable alternative

8.37 Solvent squeeze bottles

8.38 Desiccant

8.39 Sample Refrigerator, capable of maintaining temperature >0°C and ≤10°C, and capable of being secured with access limited to approved staff

8.40 Standards Refrigerator(s)

8.41 Reagents and Samples

8.41.1 Samples from sample batch

8.41.2 Batch Sample

8.41.2.1 Deionized Water, ASTM Type I

8.41.2.2 Acetone, pure (99.9%), or as pure as can be reasonably obtained

8.41.2.3 Methanol, pure (99.8+%), or as pure as can be reasonably obtained

8.41.2.4 Ethanol, 200 proof

8.41.2.5 Sodium chloride, ACS grade, 99% minimum

8.41.3 Control/check stock for required analyses

8.41.4 1-Methoxy-2-Propanol (MPA), 99+%, dry

8.41.5 Hexane, anhydrous, 95% n-hexane or better

8.41.6 Acetone, ACS grade or better

8.41.7 Methanol, ACS grade or better

8.41.8 Isopropanol, ACS grade or better

9 Procedure

9.1 Aliquoting samples

9.1.1 Labeling vials

9.1.1.1 Label a 40mL amber vial with the sample ID number for each sample. This vial is for the archive aliquot.

9.1.1.2 Label a 20mL vial with the sample ID number for each sample and duplicate. This vial is for the sample aliquot.

9.1.1.3 Label an 8mL vial and a 2mL autosampler vial with the sample ID number for each sample aliquot (including the BS and duplicate aliquot). These vials are for the sample dilutions.

9.1.1.4 Label an 8mL vial and a 2mL autosampler vial for a solvent blank.

9.1.1.5 Label an 8mL vial and a 2mL autosampler vial for any control/check standards.

9.1.2 Retrieve samples from the sample refrigerator and ensure samples are at room temperature before further processing.

Shaving gels are an exception. The sample, sample aliquot, and archive

aliquot must remain refrigerated (>0°C and ≤10°C) prior to and immediately after use.

9.1.3 For an aerosol sample, be sure to follow procedures outlined in SAS05 for propellant collection and can weights prior to aliquoting the non-propellant portion.

9.1.4 Mix sample as much as possible to ensure homogeneity.

9.1.5 Open sample container using tools or implements, as necessary.

9.1.6 Some samples may require additional processing to extract the non-aerosol sample or the non-propellant portion of an aerosol sample from the delivery system such as:

9.1.6.1 Dryer Sheets

For the handling of dryer sheets, refer to APPENDIX A.

9.1.6.2 Wipes

Wring out heavily saturated wipes over a beaker or dish to collect the sample. Ensure the beaker or dish is of adequate size.

Wipes that are not heavily saturated may require the use of a syringe to facilitate sample collection. Put wipes into a 20mL or 60mL syringe to press out the sample. Repeat as necessary until archive and sample vials are filled or all wipes have been processed. At times it may be necessary to use compressed gas to assist in the pressing (refer to APPENDIX B).

Sampled and un-sampled wipes shall be stored with empty sample containers and packaging until release of custody to the client.

9.1.6.3 Liquid or Gel Beads, Solid Gels

Transfer the beads/gel to a 20mL or 60mL syringe and break apart by pressing them through the tip of the syringe. Repeat as necessary until archive and sample vials are filled or the entire sample has been processed.

9.1.6.4 In instances not addressed by section 9.1.6.1 – 9.1.6.3, where separation of the non-aerosol sample or the non-propellant portion of an aerosol sample from the delivery system is not possible, no sample dilution is prepared. Report only qualitative data from SAS05, SAS06, and SAS13.

9.1.7 Ensure the sample is a homogenous mixture, if not, mix using a stirring rod.

9.1.8 Portion the sample into a 40mL vial for an archive aliquot, and a 20 mL vial

for a sample aliquot.

9.1.8.1 Store archive aliquots in sample refrigerator.

9.1.8.2 Retain archive aliquots for three years from completed analysis. After three years, archive aliquots should be segregated/disposed of by chemical category as described in Section 7.1 for samples.

9.1.9 Dispose of excess sample as described in Section 7 of this SOP.

9.1.10 Store empty sample containers in a secure location until release of custody to the client.

9.2 Preparing Batch Sample stock. Other quantities may be prepared using the same ratios.

9.2.1 To prepare the Batch Sample stock, weigh 300g of deionized water and 50g each of sodium chloride, acetone, methanol, and ethanol into a 500mL volumetric flask. Dissolve sodium chloride completely in the deionized water prior to adding the acetone, methanol, and ethanol. Mix by inversion.

9.2.2 Fill 16mL vials with approximately 4.5mL aliquots of the Batch Sample stock solution and cap.

9.2.3 Label each Batch Sample stock vial with “Batch Sample”, preparation date, expiration date, and the preparer's initials. The expiration of the Batch Sample stock must not exceed the expiration dates of the chemicals used or three years, whichever is earliest.

9.2.4 Store Batch Sample stock aliquots in standards refrigerator (stored aliquots may be used, it is not necessary to prepare a new Batch Sample stock for every sample batch).

9.3 Prepare sample dilutions for analysis under Method 310.

9.3.1 Sample dilutions are prepared using MPA as the solvent.

There are some instances where another solvent may be required for a particular analysis as MPA may interfere with the compound of interest (e.g., trichloroethylene). In these cases, in addition to the MPA dilutions, an additional set of dilutions will be required for the affected samples. Note that all blanks, controls, calibrators, and samples for that particular analysis are to be prepared using this same alternate solvent.

9.3.2 Prepare solvent blank by filling the appropriately labeled 8mL vial and 2mL autosampler vial with the same solvent used to make the sample dilutions. Cap the vials.

9.3.3 Prepare a 25mg/mL control/check standard for any required analyses by diluting 1.0mL of the 250mg/mL stock solution into a 10mL volumetric flask and bring to volume with solvent. Transfer the control/check standard into appropriately labeled 8mL vials and 2 mL autosampler vials and cap.

9.3.4 For each sample aliquot, weigh approximately 1.0mL into a 10mL volumetric flask and record the value in LIMS. This is done for all sample aliquots, which include the Batch Sample, and duplicate aliquot.

9.3.4.1 For procedure details related to sample dilutions prepared from dryer sheets, refer to APPENDIX A.

9.3.4.2 Ensure data transfer software is open on the laboratory work station.

9.3.4.3 Ensure the accuracy of the analytical balance.

Perform a balance control on the analytical balance prior to use.

Using the forceps included with the weight set, place a 1.0g mass on the analytical balance. When the reading becomes stable, as indicated by the analytical balance, record the weight in LIMS using the Dilution Weights and Dryer Sheet Weights application. If the weight is not within the control limits (±2sd of the target value), no sample dilutions are prepared. Re-weigh the mass and record in LIMS. If the weight is still outside the control limits, there may be a problem with the analytical balance or the mass. Contact vendor for service.

9.3.4.4 Open the data collection software located on the laboratory work station desktop. Save the dilution spreadsheet under a naming system that includes the sample ID numbers. Enter the current date, analyst name, and sample batch information. Enter in the sample ID for each sample in the Sample ID column.

9.3.4.5 For each sample dilution, tare a 10mL volumetric flask on the analytical balance (it is acceptable to add a small amount of MPA (1mL – 5mL), to the flask prior to this step). Pipette approximately 1mL of sample aliquot into the tared flask.

An equally proportioned dilution of less volume may be necessary if sample size is limited. In these cases, note the volumes used (the sample volume pipetted and the size of the volumetric flask) and be sure to adjust the sample dilution weight entered into LIMS to account for the amount used.

For samples that cannot be pipetted (such as viscous liquids, creams, pastes, gels, and semi-solids), a transfer tube may be used in place of a pipettor, or the sample may be dispensed into the flask directly from the

sample container.

9.3.4.6 Weigh and transfer the weight value to the dilution spreadsheet.

9.3.4.7 Dilute to 10mL with MPA. Invert or vortex to mix (a homogenizer or sonicator may also be used). Samples may require filtering/settling. Make note of any samples that do not dissolve well on the dilution spreadsheet.

Indications of sample dilutions requiring filtering or settling include cloudiness, floating particles, undissolved solids, and gel like appearance.

9.3.4.8 Transfer the sample dilutions into appropriately labeled 8mL vials and 2 mL autosampler vials and cap.

When a sample dilution requires filtering, attach a syringe filter to the tip of a 3mL disposable syringe and pass the sample dilution through the filter into appropriately labeled 8mL vial and 2mL autosampler vials and cap.

If the sample becomes turbid, repeat filtering.

9.3.4.9 Perform a Balance Check after all weighing is complete.

Using the forceps included with the weight set, remove the 1.0g mass and place it on the analytical balance. When the reading becomes stable as indicated by the analytical balance, record the weight in LIMS using the Dilution Weights and Dryer Sheet Weights application.

If the weight is not within the control limits, all sample dilutions are invalid. Prepare all sample dilutions again as they must be bracketed between a successful Balance Control and Balance Check.

9.3.4.10 Upload data for sample dilutions to LIMS using the Dilution Weights and Dryer Sheet Weights application

10 Quality Control

10.1 Quality Controls

QC TYPE	FREQUENCY	CRITERIA	CORRECTIVE ACTION
Balance Control	Daily prior to use	±2sd of the target value	If outside control criteria, no sample dilutions are prepared. Re-weigh the mass and record in LIMS. If the weight is still outside the control limits, there may be a problem with the balance or the mass. Contact

QC TYPE	FREQUENCY	CRITERIA	CORRECTIVE ACTION
			appropriate personnel for service.
Balance Check	After weighing session	±2sd of the target value	If outside the control criteria, all sample dilutions are invalid. Perform the Balance Control after bringing the balance back into control. Re-prepare sample dilutions.

10.2 Equipment Requirements

10.2.1 The balances require calibration by an outside source annually.

10.2.2 The 1.0g mass is calibrated by an outside source annually.

10.2.3 Sample Refrigerator temperature shall be maintained >0ºC and ≤10ºC. Verify and record refrigerator temperature each working day.

Exceedance of temperature range for more than one week may compromise the integrity of the archive samples and requires notification to the client.

10.2.4 Pipettors require calibration by an outside source annually.

11 Sample and Data Management

11.1 Data management consists of samples logged into the LIMS, documentation of unusual occurrences and their resolutions, creation of data packages (monthly, amendments, and special projects) for peer review and management approval, submittal of data to clients, and archival procedures for sample media and respective chains of custody. Program and maintenance notebooks and/or logbooks are to be kept with the instrumentation at all times.

11.2 Sample and data management follow procedures outlined in the QCM. The LIMS Manual describes data management procedures as they pertain to LIMS for this SOP.

11.3 Retain archive aliquots for three years from completed analysis.

11.4 Information that has been designated as confidential, proprietary, or trade secrets must be maintained in a locked file cabinet in a secure area. Access to this file cabinet is subject to management approval.

12 Calculations

There are no calculations in this method.

13 References

13.1 Method 310 Determination of Volatile Organic Compounds (VOC) in Consumer Products and Reactive Organic Compounds (ROC) in Aerosol Coating Products, https://ww2.arb.ca.gov/sites/default/files/2022-10/CARB_Method310_MLD_SAS_08012022.pdf, August 1, 2022

13.2 The California Consumer Products Regulations, Title 17, California Code of Regulation, Division 3, Chapter 1, Subchapter 8.5, Article 1 – Article 5

13.3 SAS05 Standard Operating Procedure for the Determination of Compounds in Aerosol Consumer Product Propellant by Gas Chromatography, Revision 3.2, August 18, 2010 or current

13.4 SAS06 Standard Operating Procedure for the Tentative Identification of Compounds in Consumer Products by Headspace Gas Chromatography/Mass Spectrometry, Revision 1.5, October 28, 2021 or current

13.5 SAS13 Standard Operating Procedure for Consumer Product Sample Batch Management and Reporting, Revision 0.0, August 5, 2019 or current

13.6 CARB NLB Laboratory Quality Control Manual, December 7, 2021 or current

13.7 MLD076 Standard Operating Procedure for Preparation of Northern Laboratory Branch's Standard Operating Procedures, Revision 1.0, December 30, 2021, or current

13.8 California Air Resources Board, Chemical Hygiene Plan for Northern Laboratory Branch 1927 13th Street, 1900 14th Street", June 17, 2022 or current

13.9 Consumer Products Database Special Analysis Section (Oracle Database and Applications Manual for LIMS)

14 Revision History

SOP/Addendum Identification	Approval Date	Description of Change
SAS14 Revision 0.0	August 5, 2019	New SOP for Consumer Product Sample Preparation
SAS14 Revision 0.1	June 14, 2024	Editorial and administrative changes

APPENDIX A

Handling of an Atypical Sample Matrix: Dryer Sheets

1 Summary

This document describes procedures specific to the sampling and dilution preparation of consumer products with VOC embedded within a delivery substrate, such as those categorized as Fabric Softener – Single Use Dryer Product.

2 Equipment and Supplies

2.1 In addition to the supplies listed in Section 8 of SAS14, the following are necessary for this appendix.

2.1.1 Platform Shaker.

2.1.2 Circular Template, 60mm diameter.

3 Procedure

3.1 Label vials as indicated in SAS14 Section 9.1.1.

3.2 Ensure samples are at room temperature.

3.3 Ensure the accuracy of the analytical balance per Section 9.3.4.3 of SAS14.

3.4 Ensure data transfer software is open on the laboratory work station.

3.5 Open the data collection software located on the laboratory work station desktop. Save spreadsheet under a naming system that includes the sample ID numbers. Navigate to the “DryerSheet” spreadsheet. Enter the current date, analyst name, and sample batch information. Enter in the sample ID for each dryer sheet weight under the Sample ID column.

3.6 Take a single dryer sheet from the middle of the box of dryer sheets and weigh to the nearest 0.00001g. Transfer the whole sheet weight in the “Whole Sheet Wt (g)” column.

3.7 Using circular template cut a section from the center of the folded dryer sheet to create a sample aliquot. Weigh to the nearest 0.00001g. Transfer the sample aliquot weight in the “Aliquot Wt (g)” column.

3.8 Insert the sample aliquot into the 20mL vial, add volumetrically 10mL of MPA, and cap vial.

3.9 Perform a Balance Check after all weighing is complete per section 9.3.4.9 of SAS14.

3.10 Save and upload dilution data on the spreadsheet for dryer sheet samples to the LIMS using the Dilution Weights and Dryer Sheet Weights application.

3.11 Place the vial on its side on a platform shaker. Shake for 16 to 24 hours at 100 to 150 rpm.

3.12 Allow any particulates to settle out before transferring, filtering if necessary using a syringe and syringe filter.

3.13 Transfer each sample dilution into labeled 8mL and 2mL autosampler vials and cap.

APPENDIX B

Handling of an Atypical Sample Matrix: Wipes

1 Summary

This document describes a procedure for the sampling of wipes using the assistance of compressed gas.

2 Equipment and Supplies

2.1 In addition to the supplies listed in Section 8 of SAS14, the following are necessary for this appendix:

2.1.1 Wipe Compression Assembly (Figure 1).

2.1.2 Compressed air.

3 Procedure

3.1 Set up the apparatus.

3.1.1 Establish a flow of compressed air to the wipe compression assembly not to exceed 40psi.

3.2 Prepare archive aliquot and sample aliquot.

3.2.1 Remove plunger from 60mL syringe.

3.2.2 Insert 1-3 wipes into syringe.

3.2.2.1 If initial attempt to obtain sample from wipe fails, try a different number of wipes.

3.2.3 Insert plunger into syringe without pressing down on wipes.

3.2.4 Place archive or sample vial under the platform in the wipe compression assembly.

3.2.5 Place syringe on the platform so the syringe tip is through the assembly's center hole and the air cylinder piston is in contact with the plunger, ensuring the syringe tip is over the vial opening.

3.2.6 Actuate the air cylinder piston and adjust pressure to 25-30psi so that the air cylinder piston is activated forcing the plunger down.

3.2.7 Ensure sample collection in vial.

3.2.7.1 Increase pressure as necessary or use a different number of wipes placed in the syringe to achieve sample collection.

3.2.8 When sample flow stops, deactivate the air cylinder piston, and remove syringe from the platform.

3.2.9 Remove the syringe plunger and use forceps to remove the wipes from the syringe.

3.2.9.1 Wipes shall be stored with empty sample containers and packaging until release of custody.

3.2.10 Repeat as necessary to fill archive aliquot and sample aliquot vials.

3.3 Shutting down the apparatus.

3.3.1 Turn off the flow of compressed air.

3.3.2 Ensure the air cylinder plunger is all the way up.

FIGURE 1

WIPE COMPRESSION ASSEMBLY