Standard Operating Procedure for Consumer Product Sample Batch Management and Reporting

SAS13
Revision 0.1

Northern Laboratory Branch
Monitoring and Laboratory Division

Approval Signatures	Approval Date
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1 Introduction

This procedure describes sample batch management processes and data reporting through multiple analyses that are required under Method 310 and the California Consumer Products Regulations.

2 Summary of Method

This procedure describes creating a sample batch and carrying it through multiple processes and analyses to determine Volatile Organic Compounds (VOC) and Reactive Organic Compounds (ROC) under Method 310. Additionally, it details the process for compiling data and results for multiple analyses, generating reports for each sample, and quality control data for the sample batch.

3 Acronyms and Definitions

Acronym or Term	Definition
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.
Batch Sample (BS)	A laboratory prepared sample aliquot of known concentration for QC evaluation under Method 310.
COC	Chain of Custody (Laboratory Request and Sample Transfer Form)
duplicate	A second analysis of a sample submitted for analysis under Method 310.
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.
H&SC	Health and Safety Coordinator
HVOC	High Volatility Organic Compound
ID	Identification
LIMS	Laboratory Information Management System
LIMS Manual	Consumer Products Database Special Analysis Section (Oracle Database and Applications Manual for LIMS)
LVP	Low Vapor Pressure
LVP-VOC	Low Vapor Pressure-Volatile Organic Compounds
MVOC	Medium Volatility Organic Compound

Acronym or Term	Definition
NLB	Northern Laboratory Branch
PPE	Personal Protective Equipment
PWMIR	Product-weighted maximum incremental reactivity
QC	Quality Control
QCM	Quality Control Manual
ROC	Reactive Organic Compound(s)
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.
SOP	Standard Operating Procedure
TIC	Tentatively Identified Compound
VOC	Volatile Organic Compound(s)

4 Interferences

4.1 Interferences for each individual method are described in the method-specific SOP.

4.2 Consumer product packaging may prevent separation of the product from the delivery system. In these cases, a Qualitative Results Report containing only qualitative data will be provided to the client.

4.3 Consumer product sample matrix may be such that no or only limited analysis is possible. In these cases, note this in the Comments Section on the report to the client.

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 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 are not limited to clothing for a low temperature environment, non-slip footwear, and hearing protection.

7 Hazardous Waste

For all 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's 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 being moved to the main hazardous waste storage area or transferred 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 in a secure location until release of custody to the client.

8 Equipment, Supplies, and Chemicals

8.1 Personal Computer

8.2 Laboratory Information Management System (LIMS)

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

9 Procedure

9.1 Determine the number of samples to analyze and obtain COC paperwork.

9.2 Use the Sample Login Data application in LIMS to create a sample batch.

9.2.1 Print a Tracking Sheet for the sample batch.

9.2.2 Log the duplicate(s).

9.2.2.1 The Tracking Sheet will have a LIMS assigned duplicate(s).

The analyst may reassign the duplicate(s) if there is not enough sample to analyze as a duplicate. Reassigning the duplicate(s) may become necessary due to sample matrix or when issues arise during analysis that result in reporting only qualitative data.

9.2.2.2 Log the duplicate(s) sample ID into the LIMS database.

9.2.3 Log the Batch Sample into LIMS (prepared in SAS14).

9.2.4 Add samples to the sample batch in LIMS.

9.3 Transfer custody of samples.

9.3.1 Fill out the Transfer Record on the COC. Include the full names of persons involved in the transfer and the date of the transfer.

9.3.2 The analyst may take custody of samples within the sample batch at different dates and times.

9.4 Using the Headspace, Ingredients, and Report Comments application, record the sample ingredients into LIMS. If a sample does not have an ingredient list, note this in LIMS. A sample ingredient list is not required prior to beginning sample analysis; however, it can be helpful in determining what analyses are necessary.

9.5 Perform propellant analysis for aerosol samples in the sample batch following SAS05.

9.6 Prepare non-aerosol samples and the non-propellant portion of aerosol samples in the sample batch for analysis following SAS14.

9.7 Analyze the sample batch utilizing the following SOPs: SAS01, SAS03, SAS04, SAS06, and SAS07 (only ACETONE).

9.8 Use information obtained in SAS06 and sample ingredients to assist in determining the need for additional analyses (e.g., if there is a tentatively identified compound (TIC) or ingredient that is quantitated in another SOP). Analyze the sample batch utilizing the following SOPs as necessary: SAS02, SAS07, SAS09, and SAS15.

9.9 Report comments

Use the Headspace, Ingredients, and Report Comments application to input any general comments into LIMS (e.g., any management-approved procedure deviations).

9.10 Use the Gravimetric Analysis application in LIMS to edit the use of gravimetric data when samples contain LVP-VOC.

9.11 Report generation

9.11.1 Use the Consumer Products Reports application in LIMS to generate reports for all samples in the sample batch including the duplicate. Select the appropriate report type for each sample based on the requirements in the Consumer Products Regulations. If only qualitative data is being reported for a sample, generate a Qualitative Results Report in lieu of another report type.

9.11.2 Use the Consumer Products Reports application in LIMS to generate a Batch Sample Report.

9.11.3 Use the Consumer Products Reports application in LIMS to generate a Consumer Products Sample Batch Results and QC Summary report.

9.12 Use the Consumer Products Reports application in LIMS to submit data packets and reports for peer review, and management review and approval.

The NLB QCM outlines the review process and criteria.

10 Quality Control

10.1 Quality Controls

QC TYPE	FREQUENCY	CRITERIA	CORRECTIVE ACTION
Duplicate	One duplicate per ten or fewer samples in the sample batch	Absolute difference between the duplicate and the corresponding sample must be less than 3 percent VOC.	If outside the QC criteria, sample results are invalidated. Reanalyze the sample batch. Reassigning the duplicate may be necessary if sample matrix is an issue.
Batch Sample	One per sample batch	±3 percent VOC of the known value.	If outside the QC criteria, sample results are invalidated. Reanalyze the sample batch.

10.2 Equipment Requirements

10.2.1 Sample Refrigerator temperature shall be maintained >0ºC and ≤10ºC. Verify and record refrigerator temperatures 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.

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 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

12.1 LIMS automatically performs all calculations.

12.2 The average of the water results from SAS03 and SAS04 will be calculated and used for the purpose of VOC calculations. In cases where only one result is used in the calculation of VOC, a justification shall be documented in LIMS and on the report to the client.

$$\text{weight fraction of water (H)} = \frac{(H_{SAS03} + H_{SAS04})}{2}$$

Where:

H_{SAS03} = sample result in weight fraction from SAS03

H_{SAS04} = sample result in weight fraction from SAS04

12.3 Absolute difference (AD):

$$AD = |\text{sample result} - \text{duplicate result}|$$

12.4 Calculation of VOC content

12.4.1 Article 1. Antiperspirants and Deodorants

This section specifies the equations that shall be used to calculate the Medium Volatility Organic Compound (MVOC) and High Volatility Organic Compound (HVOC), of consumer products under section 94500, which shall be reported as percent by weight.

12.4.1.1 Aerosol Products

12.4.1.1.1 The following equations shall be used to calculate the HVOC of aerosol products, which shall be reported as percent by weight:

$$\% \text{ HVOC} = \left[\sum_{i=1}^{h}\left(\frac{HV}{WL + WP}\right)_i\right] \times 100$$

Where:

HV = weight of HVOC compound (g), in product.

WL = weight in grams (g) of a non-aerosol sample or the non-propellant portion of an aerosol sample, excluding container and packaging.

WP = weight (g) of propellant.

h = number of HVOC compounds identified.

12.4.1.1.2 The following equations shall be used to calculate the MVOC of aerosol products, which shall be reported as percent by weight:

$$\% \text{ MVOC} = \left[\sum_{i=1}^{m}\left(\frac{MV}{WL + WP}\right)_i\right] \times 100$$

Where:

MV = weight of MVOC compound (g), in product.

m = number of MVOC compounds identified.

12.4.1.2 Non-Aerosol Products

12.4.1.2.1 The following equations shall be used to calculate the HVOC of non-aerosol products, which shall be reported as percent by weight:

$$\% \text{ HVOC} = \left[\sum_{i=1}^{h}\left(\frac{HV}{WL}\right)_i\right] \times 100$$

12.4.1.2.2 The following equations shall be used to calculate the MVOC of non-aerosol products, which shall be reported as percent by weight:

$$\% \text{ MVOC} = \left[\sum_{i=1}^{m}\left(\frac{MV}{WL}\right)_i\right] \times 100$$

12.4.2 Article 2. Consumer Products

This section specifies the equations that shall be used to calculate the VOC content of a product.

12.4.2.1 Aerosol Products

12.4.2.1.1 For aerosol products, except those containing LVP-VOC, the percent VOC content shall be calculated using the following equation:

$$\% \text{ VOC} = \left[\frac{WL\ (\ TV - A - H - EL\ ) + (WP - EP)}{WL + WP}\right] \times 100$$

Where:

WL = weight (g) of a non-aerosol sample or the non-propellant portion of an aerosol sample, excluding container and packaging.

TV = weight fraction of total volatile content in a non-aerosol sample or in the non-propellant portion of an aerosol sample.

A = weight fraction of ammonia (as NH_4^+) in a non-aerosol sample or in the non-propellant portion of an aerosol sample.

H = weight fraction of water in a non-aerosol sample or in the non-propellant portion of an aerosol sample.

EL = weight fraction of exempt compound(s) in a non-aerosol sample or in the non-propellant portion of an aerosol sample.

WP = weight (g) of propellant.

EP = weight (g) of exempt compound(s) in propellant.

12.4.2.1.2 For aerosol products containing LVP-VOC, the percent VOC content shall be calculated using the following equation:

$$\% \text{ VOC} = \left[\frac{WL\ [\ (1 - H) \times (\ 1 - LVP\ ) - EL\ ] + (WP - EP)}{WL + WP}\right] \times 100$$

Where:

LVP = weight fraction of LVP-VOC compounds and/or mixtures

in the non-propellant, non-aqueous portion.

1 – H = weight fraction of the non-propellant portion that does not contain water.

1 – LVP = weight fraction of the non-propellant, non-aqueous portion that is volatile.

Volatile compounds, such as ammonia, that do not meet the definition of a VOC in the Consumer Products Regulations will not count toward the total percent VOC content of a product.

12.4.2.2 Non-Aerosol Products

12.4.2.2.1 For non-aerosol products, that do not contain LVP-VOC, the percent VOC content shall be calculated using the following equation:

$$\% \text{ VOC} = (\text{TV} - \text{A} - \text{H} - \text{EL}) \times 100$$

12.4.2.2.2 For non-aerosol products containing LVP-VOC, the percent VOC content shall be calculated using the following equation:

$$\% \text{ VOC} = [(1 - \text{H}) \times (1 - \text{LVP}) - \text{EL}] \times 100$$

12.4.2.3 For consumer products with VOC embedded within a delivery substrate, such as Fabric Softener – Single Use Dryer Product, VOC shall be calculated as total weight (g) VOC per use.

12.4.2.3.1 For those products, that do not contain LVP-VOC:

$$\text{VOC per use (g)} = (\text{TV} - \text{A} - \text{H} - \text{EL}) \times \text{TW}$$

Where:

TW = total weight (g) of VOC and delivery substrate per use, excluding container and packaging.

12.4.2.3.2 For those products containing LVP-VOC:

$$\text{VOC per use (g)} = [(1 - \text{H}) \times (1 - \text{LVP}) - \text{EL}] \times \text{TW}$$

12.5 Calculation of PWMIR using ROC content

This section specifies the equation that shall be used to calculate the PWMIR

$$\text{PWMIR} = \left[\sum_{i=1}^{r} \left(\frac{\text{RW}}{\text{WL} + \text{WP}} \right)_i \times \text{MIR}_i \right]$$

Where:

RW = weight of ROC compound and/or hydrocarbon solvent (g) in product.

r = number of ROC compounds and hydrocarbon solvents identified.

MIR = maximum incremental reactivity (MIR) value, as stated in Title 17, CCR, sections 94700 and 94701.

WL = weight (g) of a non-aerosol sample or the non-propellant portion of an aerosol sample, excluding container and packaging.

WP = weight (g) of propellant.

13 References

13.1 Method 310 Determination of Volatile Organic Compounds (VOC) in Consumer Products and Reactive Organic Compounds (ROC) in Aerosol Coating Products, 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 SAS01 Standard Operating Procedure for the Total Volatile Measurement of Consumer Products, Revision 1.7, October 24, 2018

13.4 SAS02 Standard Operation Procedure for the Measurement of Ammonium Ion in Aqueous Consumer Products Using Ion Chromatography, Revision 3.0, November 17, 2021

13.5 SAS03 Standard Operating Procedure for the Karl Fischer (KF) Determination of Water with KF Drying Oven in Consumer Products, Revision 3.0, September 19, 2012

13.6 SAS04 Standard Operating Procedure for Water Determination in Consumer Products Using Gas Chromatography, Revision 1.7, October 28, 2021

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

13.8 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

13.9 SAS07 Standard Operating Procedure for the Determination of Exempt and Non-Exempt Compounds Generally Found in Consumer Products by Gas Chromatography-FID, Revision 1.9, August 28, 2012

13.10 SAS09 Standard Operating Procedure for the Determination of Boiling Point Distribution in Consumer Products by Gas Chromatography, Revision 1.5, February 9, 2022

13.11 SAS14 Standard Operating Procedure for Consumer Product Sample Preparation, Revision 0.0, August 5, 2019

13.12 SAS15 Standard Operating Procedure for the Determination of Compounds in Aerosol Coating Consumer Products Using Gas Chromatography, Revision 0.0, August 5, 2019

13.13 NLB Laboratory Quality Control Manual, December 7, 2021

13.14 MLD076 Standard Operating Procedure Preparation of Northern Laboratory Branch's Standard Operating Procedures, Revision 1.0, December 30, 2021

13.15 Chemical Hygiene Plan for Northern Laboratory Branch, 1927 13th Street, 1900 14th Street, July 19, 2023 (or most current version)

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

14 Revision History

SOP/Addendum Identification	Approval Date	Description of Change
SAS13 Revision 0.0	August 5, 2019	New SOP for sample batch management and reporting under Method 310.
SAS13 Revision 0.1	November 5, 2024	Editorial and administrative changes.