State of California
California Environmental Protection Agency
Air Resources Board
MLD/QMS-066 (NEW 4/14)

QUALITY MANAGEMENT DOCUMENT ADDENDUM

(District completes Sections 1 through 6 -- please type)

Section 1. ARB Document
☐ Quality Management Plan (QMP)
☐ Quality Assurance Project Plan (QAPP)
☒ Standard Operating Procedure (SOP)

Section 2. District Information		
District Name:	Northern Laboratory Branch Monitoring and Laboratory Division	
District Address:	1900 14th Street, Sacramento	
District Contact Name/Phone Number:	Keith Kennedy	916-322-2496
District Signature/Date:	Keith Kennedy	6/20/25

Section 3. Document Title (*specify exact title, revision #, and date of ARB Document(s) that your District proposes to modify)*	**Date**
Standard Operating Procedure for the Tentative Identification of Compounds in Consumer Products by Headspace Gas Chromatography/Mass Spectrometry SAS06 Revision 1.5	October 28, 2021

Section 4. Proposed Deviation(s)
(specify exact section(s), page number(s) and language in existing ARB document that your District proposes to modify and then specify proposed modification (including any spreadsheets or forms).

Current Language (Page 3 Section 8.1.1):
Headspace sampler (e.g., Agilent G1888 or Agilent 7697A)

Proposed Language (Page 3 Section 8.1.1):
Headspace sampler (e.g. Agilent G1888, Agilent 7697A, or equivalent)

Current Language (Page 4 Section 9.1.3):

GC cycle time	43 min

Proposed Language (Page 4 Section 9.1.3):

GC cycle time (7697A)	43 min
GC cycle time (8697)	39 min

Current Language (Page 4 Section 9.1.3):

Tune File	atune.u

Proposed Language (Page 4 Section 9.1.3):

Tune File (7697A)	atune.u
Tune File (8697)	etune.u

Current Language (Page 4 Section 9.1.3):

Vial Pressure	17 psi

Proposed Language (Page 4 Section 9.1.3):

Vial/Fill Pressure	17 psi

Current Language (Page 4 Section 9.1.3):

Injection	0.2 min

Proposed Language (Page 4 Section 9.1.3):

Injection (duration)	0.2 min

Current Language (Page 4 Section 9.1.3):

Loop Fill	default

Proposed Language (Page 4 Section 9.1.3):

Loop Fill (mode)	default

Current Language for Loop Size (Page 4 Section 9.1.3):
None currently specified.

Proposed Language for Loop Size (Page 4 Section 9.1.3):

Loop Size	1 mL

Current Language (Page 4 Section 9.1.3):

Shaking	50/min

Proposed Language (Page 4 Section 9.1.3):

Vial Shaking	Level 4; 50 shakes/min at 180 cm/s^2

Section 5. Justification for Deviation(s) *(provide explanation of why modification(s) to existing ARB document is necessary)*
The changes above reflect: • Equipment clarification to promote future optimized laboratory productivity. • Clarification and correction of some points.

Section 6. Attachment(s) ☐ *(specify attachment titles and number of pages, include modified spreadsheets or forms)*	**# of Pages**

Section 7. ARB Approval *(completed by ARB)*		
Name/Phone Number:	Manisha Singh, Ph.D.	279-208-7896
Title:	Chief, Quality Management Branch	
Signature/Date:	Manisha Singh	6/18/2025
Addendum Number	A55	

Completed form must be scanned/emailed or mailed to:

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Standard Operating Procedure for the Tentative Identification of Compounds in Consumer Products by Headspace Gas Chromatography/Mass Spectrometry

SAS06
Revision 1.5

Northern Laboratory Branch
Monitoring and Laboratory Division

Approval Signatures	Approval Date
Manisha Singh, Ph.D., Chief Quality Management Branch	August 23, 2021
Michael Werst, Chief Northern Laboratory Branch	10/28/2021

Disclaimer: Mention of any trade name or commercial product in this standard operating procedure does not constitute endorsement or recommendation of this product by the California Air Resources Board. Specific brand names and instrument descriptions listed in the standard operating procedure are for equipment used by the California Air Resources Board's laboratory. Any functionally equivalent instrumentation is acceptable.

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Standard Operating Procedure for the Tentative Identification of Compounds in Consumer Products by Headspace Gas Chromatography/Mass Spectrometry

1 Introduction

This standard operating procedure (SOP) describes a qualitative procedure for tentatively identifying compounds present in a non-aerosol or the non-propellant portion of an aerosol consumer product. This SOP aids in determining what analyses to complete under Method 310 as required by the Consumer Products Regulations. Development of this SOP was aided by procedures specified in US EPA Method 8240B.

2 Summary of Method

Approximately 100 µL of the sample aliquot is introduced into a 20 mL headspace vial containing 4.0 mL of dispersant. The vial is then sealed and placed into the headspace sampling unit. The dispersant is used to ensure that small variations in sample polarity will not affect the analyte(s) partition coefficient. The vial is equilibrated to 80ºC, pressurized to a constant pressure, and an aliquot of the headspace gas is injected into a Gas Chromatograph/Mass Spectrometer (GC/MS).

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.
ASTM	American Standards for Testing and Materials
Base peak	The most intense (tallest) peak in a mass spectrum.
CARB	California Air Resources Board
dispersant	10% deionized water/90% polyethylene glycol 400
GC/MS	Gas Chromatograph/Mass Spectrometer
HS	Headspace
He	Helium
i.d.	inner diameter
ID	Identification
LIMS	Laboratory Information Management System
LIMS Manual	Consumer Products Database Special Analysis Section (Oracle Database and Applications Manual for LIMS)
MLD	Monitoring and Laboratory Division

Acronym or Term	Definition
MS	Mass Spectrometer
NLB	Northern Laboratory Branch
NIST	National Institute of Standards and Technology
PEG 400	polyethylene glycol 400
QC	Quality Control
QCM	Quality Control Manual
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.
SAS	Special Analysis Section
solvent blank	A blank consisting of reagent(s), without the target compound(s), analyzed to determine interferences or contamination during analysis.
SOP	Standard Operating Procedure
US EPA	United States Environmental Protection Agency
VOC	Volatile Organic Compound

4 Interferences

4.1 The headspace vial must be checked after sealing to ensure leakage does not occur.

4.2 Cross contamination and/or carryover may occur whenever high-level and low–level samples are analyzed sequentially. Samples where contamination and/or carryover is suspected may be reanalyzed, or another analysis may be performed to confirm the compounds present. Additionally, the analyst may run solvent blanks between samples if they suspect contamination and/or carryover may occur.

4.3 Co-elution of compounds can occur resulting in poor resolution.

5 Personnel Qualifications and Training

5.1 Prior to performing this method, new personnel must be trained by staff with expert knowledge of this method. Personnel must be trained to understand the program's requirements per any applicable State and federal regulations and 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 Plans.

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 operational (i.e., adequate ventilation).

6.4 Follow safe handling practices for compressed gas cylinders.

6.5 The GC/MS has heated areas and parts (i.e., GC oven and inlet) that can exceed 250°C. Use caution when operating the instrument.

6.6 The vial should never be heated to a temperature exceeding the boiling point of the dispersant (approximately 100°C) as this may cause a rapid increase in pressure within the vial.

7 Hazardous Waste

7.1 Dispose of headspace vial contents in the hazardous waste bottle in the satellite hazardous waste accumulation area. Dispose of satellite hazardous waste contents in accordance with the NLB Chemical Hygiene Plan.

8 Equipment, Supplies, and Chemicals

8.1 Gas Chromatographic System

8.1.1 Headspace sampler (e.g., Agilent G1888 or Agilent 7697A)

8.1.2 GC/MS system (e.g., Agilent 6890N/5973, 7890A/5975C)

8.1.3 GC column: DB-624, 60 m x 0.25 mm i.d. with 1.4 µm film, or equivalent

8.1.4 GC liner: ultra inert, straight, 2 mm i.d., or equivalent

8.1.5 Laboratory work station and software with mass spectral library database

8.2 Laboratory Information Management System (LIMS)

8.3 Volumetric flask, 1000 mL

8.4 Graduated cylinder, 100 mL

8.5 Headspace vials, 20 mL with crimp-top caps

8.6 Crimper and de-crimper for vial caps

8.7 Pipettors, capable of pipetting 100 µL and 4.0 mL volumes with pipette tips

8.8 Disposable transfer tubes, 3-5 mL capacity

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

8.10 Task wipes (e.g., Kimwipes)

8.11 Gloves, non-powdered nitrile or suitable alternative

8.12 Reagents and samples

8.12.1 Dispersant – 10% Deionized water/ 90% PEG 400

8.12.1.1 Deionized water, ASTM Type I

8.12.1.2 PEG 400

8.12.2 Sample aliquots prepared in SAS14

8.12.3 Helium, ultrahigh purity (Grade 5)

9 Procedure

9.1 Instrument Preparation

9.1.1 Ensure there is sufficient carrier gas (He) for the run.

9.1.1.1 Change the tank when the pressure regulator indicates 500 psi or less.

9.1.1.2 Ensure the output of He leaving the tank is sufficient to maintain the flows required for the method.

9.1.2 Prepare the headspace sampler: Fill solvent rinse vials with solvent, and ensure waste vials are empty.

9.1.3 Load the method in the GC/MS software and verify the following conditions:

GC/MS Parameters	
Inlet	
Mode	Split
Heater	250°C
Split ratio	45.5:1

GC/MS Parameters	
Gas type	Helium
Column	
Mode	Constant flow
Flow	1.0 mL/min
Oven	
Equilibration time	0.25 min
Initial temp	40°C
Initial hold time	8 min
Ramp 1 Rate	10°C/min
Ramp 1 Final temp	250°C
Ramp 1 Hold time	5 min
Total run time	34 min
GC cycle time	43 min
Maximum temp	260°C
Aux temp (MSD Transfer Line)	280°C
MS Parameters	
Tune File	atune.u
Acquisition Type	scan
MS Source	230°C
MS Quad	150°C
Solvent Delay	3.00 min
Start Mass (m/z)	30.0
End Mass (m/z)	400.0
Threshold	150
Scan speed	N=2
Headspace System Parameters	
Temperature	
Vial oven temperature	80°C
Loop temperature	120°C
Transfer line temperature	180°C
Pressure	
Transfer gas flow	Controlled by GC carrier gas flow; flow should be set to > 30 mL/min for instruments utilizing manual pressure controls
Vial pressure	17 psi
Timing	
Injection	0.2 min
Loop equilibration	0.01 min
Loop fill	default
Vial equilibration	20 min
Vial pressurization	default
Other	

GC/MS Parameters	
Multiple headspace extraction	OFF
Shaking	50/min

9.2 Create a sequence.

9.2.1 Begin the sequence with a solvent blank, followed by samples.

9.2.2 Verify or input the following parameters in the sequence:

Sample location
Sample names
Sample type (i.e., sample)
Method name
Tray name
Data path
Method path

9.2.3 Verify analyst initials are associated with the analytical sequence.

9.2.4 Save and print the sequence.

9.3 Analysis Preparation

9.3.1 Label headspace vials for all samples and solvent blank(s).

9.3.2 Prepare solvent blank(s) by pipetting 4.0 mL of dispersant into blank vial(s) and cap.

9.3.3 Prepare samples by pipetting 4.0 mL of dispersant into sample vials.

9.3.4 Mix sample aliquot to ensure homogeneity, vortexing if needed. Add approximately 100 µL of the sample aliquot (by pipetting or using a transfer tube) into the appropriately labeled headspace vial containing the dispersant and cap the vial. Repeat for all samples in the analytical batch.

9.4 Headspace Analysis

9.4.1 Place the vials in the headspace sampler matching vial locations to the sequence.

9.4.2 Run the sequence.

9.4.3 Upon sequence completion:

9.4.3.1 Remove vials from headspace sampler.

9.4.3.2 Verify the instrument is in standby mode.

9.5 Data Analysis

9.5.1 Open the “Enhanced Data Analysis” window.

9.5.2 For each data file, load the file and perform the following:

9.5.2.1 Print Chromatogram

9.5.2.2 For peaks of interest, determine possible spectrum matches from the mass spectral library database (e.g., NIST). Compare the library spectrum with the spectrum of the peak of interest. Print the library search results for the spectrum match.

9.5.3 Enter data into LIMS (refer to LIMS Manual: Headspace Results Entry).

10 Quality Control

10.1 Table of quality controls

QC TYPE	FREQUENCY	CRITERIA	CORRECTIVE ACTION
Solvent Blank	At minimum once at the start of the sequence	For any tentatively identified compound reported, the base peak in the solvent blank must be less than 10 % of the base peak in the sample.	For any tentatively identified compound, if the base peak in the solvent blank is greater than or equal to 10 % of the abundance of the base peak in the sample, the data for that tentatively identified compound may not be used for reporting purposes, unless confirmed by another analytical method.

10.2 Pipettors require certification 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. Additional SOPs that cover sample and data management as they pertain to sample preparation and data reporting under Method 310 include SAS13 and SAS14.

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

There are no calculations in this procedure.

13 References

13.1 Method 310 Determination of Volatile Organic Compounds (VOC) in Consumer Products and Reactive Organic Compounds (ROC) In Aerosol Coating Products, May 25, 2018

13.2 CARB NLB Laboratory Quality Control Manual, September 17, 2018

13.3 MLD076 Standard Operating Procedure Preparation of Northern Laboratory Branch's Standard Operating Procedures, Revision 0.0

13.4 NLB Chemical Hygiene Plan, June 2019 (or most current version)

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

13.6 SAS13 Standard Operating Procedure for Consumer Product Sample Batch Management and Reporting

13.7 SAS14 Standard Operating Procedure for Consumer Product Sample Preparation

13.8 US EPA Method 8240B, Revision 2, September 1994, Final Update IIA to the Third Edition of the Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), EPA publication SW-846

14 Revision History

	Date	Updated Revision	Original Procedure
1	**Description:** Revision 1.1		

	Date	Updated Revision	Original Procedure
	March 10, 1998	SOP MLD ES06 Revision 1. Adjusted document font to Times New Roman 12. Inserted appendix A formerly a stand-alone document.	Unknown
2	**Description:** Revision 1.2		
	February 16, 2005	MLD SOP SAS06 Revision 1.2. Adjusted document font to Arial 12. SOP updated to reflect current practices. Corrected revision enumeration.	SOP MLD ES06 Revision 1 (Revision 1.1).
3	**Description:** Revision 1.3		
	June 15, 2007	SOP updated to reflect current practices.	MLD SOP SAS06 Revision 1.2.
4	**Description:** Revision 1.4		
	August 23, 2010	SAS06 Revision 1.4. SOP updated to reflect current practices.	Unknown
5	**Description:** Revision 1.5		
	October 28, 2021	Reviewed for grammar and content, and compliance with the most recent versions of the QC Manual and MLD076 Revision 0.0. Miscellaneous additions/deletions made. Added clarification to the revision history. Changed SOP title to more accurately reflect the analysis being performed.	SAS06 Revision 1.4.