ALISO CANYON GAS LEAK

Time Critical Residential Indoor Environmental Sampling Workplan

Aliso Canyon Natural Gas Incident
Porter Ranch Community
Los Angeles, California

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1.0 INTRODUCTION AND BACKGROUND

1.1 Introduction

Since the beginning of the natural gas leak from oil and gas Well SS-25 at the Aliso Canyon storage facility in October 2015, the Los Angeles Department of Public Health (Public Health) and other local and State agencies have been actively monitoring and/or assessing the environmental and health issues related to the incident. During the 16-week period of October 28, 2015 to February 18, 2016, Public Health received approximately 700 health complaints associated with the gas leak. In the two-week period of February 18 to March 3, 2016, Public Health has received 150 health complaints from residents upon returning to their homes after the leak from Well SS-25 was officially sealed. This presents a unique public health challenge, as the cause of the reported post-sealing symptoms is unclear.

From March 10 through 12, 2016, Public Health and the California Department of Public Health (CDPH) conducted a door-to-door health assessment referred to as a CASPER, or "Community Assessment for Public Health Emergency Response" to more broadly investigate the nature of health complaints within the Porter Ranch community. CASPER is an open source epidemiologic health assessment tool prepared by the Centers for Disease Control and Prevention (CDC) to rapidly collect household-based health information about the health status and basic needs of an affected community from an environmental incident. Using the CASPER survey results from the Porter Ranch Community and the CASPER statistical methods, Public Health and emergency managers have made informed time-critical decisions to develop the scope of continued investigation into the Porter Ranch community health.

1.2 Background

The Aliso Canyon natural gas leak at Well SS-25 was an unprecedented event lasting several months, releasing approximately 94,000 metric tons of methane, and affecting the health and safety of thousands of residents in the nearby Porter Ranch community. This event is now considered the largest natural gas leak in U. S. history. Well SS-25 was officially sealed on February 18, 2016. However, there remains significant work to assure Public Health and members of the adjoining community that both indoor and outdoor environmental conditions have

returned to typical background levels for the Los Angeles area. The Aliso Canyon Well SS-25 and the affected communities are hereby considered the 'site'.

Some individual residents continue to report symptoms after returning home and have also reported that the symptoms subside after going back to their hotels. The timing of these symptoms could be indicative of a condition present in the indoor residential environment. Public Health created a map of the recently received health complaint reports, and the highest concentrations of reported symptoms appears to correlate with the location of the residences closest to and immediately downwind of Well SS-25 at the Aliso Canyon Facility (Figure 1).

Preliminary CASPER results indicate that 62% of households within a 3-mile radius of Well SS-25 report health symptoms that they think were caused by or related to the gas leak since the well was capped on February 11, 2016. The most commonly reported symptoms were headache (82%), eye/nose/throat irritation (71%-76%), cough (73%), stuffy/runny nose (69%), stress (69%), fatigue (66%), nausea (66%), and dizziness or light headedness (64%). These results suggest that environmental conditions may exist in the Porter Ranch Community which could be causing acute health issues.

In response to residents reporting continued health issues, Public Health has determined that a comprehensive indoor sampling strategy is necessary. The sampling results will also be used to help determine whether any of the environmental conditions analyzed can be directly or indirectly associated with the release of substances into the Porter Ranch Community from the Aliso Canyon Facility Well SS-25 gas leak and or subsequent well sealing operations.

2.0 DATA QUALITY OBJECTIVES

This section provides site-specific information within the context of the relevant steps of the Data Quality Objectives (DQO) process.

2.1 Statement of the Problem

The stated problem for this Residential Indoor Environmental Sampling Workplan (RIESW) is: the release of substances into the Porter Ranch Community from the Aliso Canyon Facility Well SS-25 gas leak and or subsequent well sealing operations may have resulted in a change in environmental conditions within a representative group of residences.

2.2 Purpose

The purpose of this this RIESW) is to identify potential contaminants in the indoor environment that may be associated with the gas leak.

2.3 Objective

The objective of this RIESW is to determine whether or not there are indoor environmental conditions present within residences at the Porter Ranch Community that are indicative of an adverse impact associated with the previous gas leak and or subsequent well sealing operations from the Aliso Canyon Facility.

The principal study questions are:

1. Are there concentrations of chemical compounds and/or particulate matter in the indoor air that are indicative of impacts associated with the closed gas leak and/or subsequent well sealing operations from the Aliso Canyon Facility SS-25 gas leak?

2. Are there concentrations of chemical compounds and/or particulate matter present on interior surfaces that are indicative of impacts associated with the previous gas leak and/or subsequent well sealing operations from the Aliso Canyon Facility Well SS-25 gas leak?

3. Are the collected data of sufficient quantity and quality to answer questions 1 and 2?

2.4 Information Utilized to Answer the Study Questions

The following data, site conditions, and other project information will help answer the principal study questions posed above:

Inputs to Question 1

- The air handling unit (AHU) fan of the individual residences will be activated for a minimum period of 10 minutes immediately prior to air sampling. The purpose of this is to potentially remobilize particulate matter that may have been deposited within the ducting of the AHU system. This allows for some normalization of the indoor air to conditions and is most representative of actual conditions when the residence is occupied.

- Common routes of exposure to ambient contaminants are respiration and absorption. Therefore, sampling of indoor air will provide the best profile of contaminants present, to which a homeowner could be exposed while occupying their home.

- Sampling of the indoor air environment of 100 representative residences from the group with the highest concentration of reported symptoms will provide a conservatively skewed sample group. If impacts are present, it would follow that they would be most likely to be detected in these residences.

- Utilizing specific sample collection protocols and laboratory analysis will result in representative and technically supportable data.

Inputs to Question 2

- Another route of exposure is from incidental ingestion from affected surfaces when touched and then inadvertently ingested.

- The sampling and analysis of settled particulate matter on surfaces in a home will allow for comparison with the likely composition of the gas effluent from the well and/or materials associated with the closure attempts and an assessment of what could become airborne within a home.

- Utilizing specific sample collection methods and protocols as well as laboratory analysis will result in consistent and technically supportable data.

Inputs to Question 3

- Approximately 100 residences will be included in the study to provide a representative sample of indoor environmental conditions.
- Individual test methods have been selected based on chemicals of potential concern, analytes included in a test method, and detection limits.

2.5 The Boundaries of the Study

The Target Area for the study has been selected based on the geographic location within the Porter Ranch Community identified using the following criteria:

- SCAQMD odor complaints
- Reported symptoms from DPH health complaint log
- Relocation density

The Porter Ranch Community Target Area is shown on the included Figure and is generally bound by the red and orange shaded areas, where there is a higher density of symptom reporting. The 100 representative residences selected for sampling will come from areas with the highest number of reported symptoms subject to authorization by property owners. An additional 10 residences in an un-affected area will be selected as a comparison group and sampled. Residences in the comparison group will be sampled using the same protocols to provide estimates for local background conditions.

The sampling events are anticipated to commence upon obtaining the access agreements from the selected residences. Sample collection for the particulate matter will require approximately one hour per residence. Sample collection for indoor air will require a minimum of 24 hours per residence. Collection of the particulate samples and placement of the sampling media is proposed to be conducted between 9AM and 6PM.

This RIESW specifies the sampling protocols and laboratory analysis methods that will be implemented during the performance of this study. Indoor air and particulate sampling data collected from each residence from within the target area will be compared with data obtained from the control group and other health related guidelines or criteria. Final results will be tabulated and summarized in a report upon completion of the study.

2.6 Sampling Rationale

Natural gas released during the Aliso Canyon event consists of >92% methane. However, elevated levels of methane are not persistent in typical ambient outdoor air. It is postulated that other chemicals or petroleum may have been entrained during the release and/or sealing of Well SS-25 at the Aliso Canyon Facility which could have migrated through the air into the residences of the Porter Ranch Community.

Therefore, sampling of the indoor air and surfaces of the residences is appropriate to determine if chemicals of concern are present that are related to the natural gas leak and are at levels that could be indicative of a health risk to residents.

2.6.1 Indoor air

The samples will be obtained to assess the relative concentrations of chemical constituents in the indoor air of the individual residences. Sampling will be conducted to determine detectable concentrations of: volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), total petroleum hydrocarbons (TPH), sulfur compounds, and metals. This data will be evaluated for indications of potential impacts associated with the Aliso Canyon gas leak. Sampling protocols and laboratory analysis methods are discussed in Section 4.0.

2.6.2 Indoor Surfaces

Wipe and contact (Biotape™ or similar) samples will be obtained from indoor surfaces to assess the character and composition of particulates that may be present in the residences. The samples will be obtained from horizontal surfaces in locations with visual indications of particulate that may include such as countertops, shelves, floors, etc. Only smooth, impervious surfaces, such as metals, glass, painted or laminated surfaces, coated desk surfaces, and vinyl floors, should be sampled. Wipe and/or contact sampling protocols are not applicable for porous surfaces (e.g., concrete, wood, asphalt, plasterboard), because long-term accumulation of and penetration of contaminants into the matrix may occur and all particulate may not be retrievable.

This data will be evaluated for indications of potential impacts associated with the Aliso Canyon gas leak. Sampling protocols and laboratory analysis methods are discussed in Section 4.0.

2.6.3 Field Sampling Equipment

Indoor air sampling will be performed using summa canisters, Tedlar® bags, sorbent tubes, vacuum pumps, and particulate cartridges. Surface particulate sampling will be performed using wipes and bio-tape. Specific sampling equipment, and sampling methodologies and laboratory test methods is described in detail in Section 4.0.

3.0 HEALTH AND SAFETY

In accordance with standard environmental procedures, a project specific Health and Safety Plan (HSP) describing safety aspects of the work to be performed at the site. The HSP will be prepared in compliance with the Occupational Safety and Health Administration (OSHA) regulation 29 CFR 1910.120 and California Code of Regulations 5192. The HSP will be reviewed and signed by a Certified Industrial Hygienist (CIH). The HSP will be read and signed by the field team personnel on a daily basis.

4.0 FIELD SAMPLING PROCEDURES AND LABORATORY ANALYSIS

4.1 Pre-Sampling

While the exact number and specific addresses of the residences to be sampled has not yet been finalized, the intent is to follow the same protocol for each residence including the residences in the control group from start to finish as described in the following sections. Sampling is intended to be representative of typical indoor environmental conditions of each home. Field personnel will be required to wear Personal Protective Equipment (PPE) appropriate for the types of sampling described below and at a minimum will include shoe coverings and powder free disposable nitrile gloves.

Initially, sampling personnel will arrive at the scheduled location and make contact with the owner, occupant, or representative. It is anticipated that the sampling team will meet the owner/representative outside at the curb of the residence at a time to be determined prior to the sampling event. Access to the individual properties for the purpose of sampling will be coordinated by Public Health. Interaction with or questions from the home owner such as access to rooms will be managed by Public Health officials.

A field monitoring sheet will be completed for each home that will note the name of the field staff, sample locations, sample identification names, visual signs of black particulate matter, and relative sampling information or other observations.

4.2 Air Sampling Procedures and Laboratory Analysis

Upon entering the residence, the AHU fan will be turned on for a minimum period of ten minutes to circulate the indoor air and to re-suspend any settled particulate that may be within the AHU system and/or home. The fan can remain on during the sample collection period. Indoor air samples will be collected from one central location within the residence in the downstairs living area within the breathing zone at an approximate height of 2 to 6 feet above the floor surface.

The entry to the house during sampling should be limited to the sampling investigators to reduce the potential for alteration of the existing conditions during sampling.

The sample containers/media will consist of laboratory provided 6-liter summa canister, a 1-liter Tedlar® bag, a sampling pump affixed with a sorbent tube, and another sampling pump affixed with a pre-weighed 37mm PVC filter cartridge. The methodology for collecting a sample for each type of container/media is included in the Appendix. The detection limits achievable for the sorbent tubes and the particulate filters are dependent upon the actual duration that a specific volume of air is pumped through the sample media. Generally, the greater the specific volume of air pumped, the lower detection limit. The sample duration was selected to achieve low detection limits and be representative of diurnal barometric fluctuations that may affect air density and air flow rates. The air flow rate and duration of collection, the chemical categories and test methods, and the hold time for each sample container type is summarized as follows:

Container	Flow Rate	Collection Period	Chemical Category	Test Method	Hold Time
6-Liter Summa Canister, batch certified	Flow controller set at a rate of 3.5 ml/minute	24-hours	VOCs	TO-15 Low Level	30 days
Sorbent Tube	Pump set at a rate of 4.7 liters/minute	24-hours	SVOCs	TO-13A	7 days to extraction; 30 days to analysis
PVC Cartridge with Pre-weighed filter	Pump set at a rate of 2.0 liters/minute	24-hours	Particulates and Metals	NIOSH 0500/7300 by ICP/MS	180 days
Tedlar bag	Lung sampler or vapor sampling box, variable pump rate acceptable	Grab sample	Sulfur Compounds	ASTM D5504-12	24 hours

Following the set sampling period, the sample container will be turned off, closed, or sealed as appropriate per the container/media specifications, labeled with a sample point identification number, stored for shipment in compliance with the test method and laboratory standards, logged on a chain of custody, and will be

submitted to a laboratory for chemical analysis at the end of each day. The analytical laboratories will be instructed to note analytes that are detected below the reporting limit but above the method detection limit (i.e. a J-Flag reported quantity). Rush turnaround time will be requested to receive the laboratory results as early as possible.

A minimum of 1 ambient air sample per neighborhood area per day will be obtained utilizing a 6-Liter Summa canister and analyzed for VOCs by TO-15 low level. The sample collection period will be the same as the sample period used for the indoor air samples.

4.3 Particulate Sampling Procedures and Laboratory Analysis

Surface particulate sampling will be accomplished using two different techniques; i.e., contact and wipe sampling methods. Contact sampling will be performed for the purpose of determining the physical character/ratio of the particulate (e.g., carbon, cellulose, biogenic, etc.). Wipe sampling will be performed for the purpose of determining the chemical composition of the particulate (i.g., distribution of heavy metals, SVOCs, and TPHs). The methodology for collecting both types of surface samples is included in the Appendix.

Type	**Media**	**Area**	**Chemical Category**	**Test Method**	**Hold Time**
Wipe	Lab prepared ('ghost wipe')	100 sq. cm	Metals	NIOSH 9102/7300 by ICP/MS	180 days
Wipe	3M 3500 Carbon Filter	100 sq. cm	Hydrocarbons	NIOSH 1500 by GC/MS	14 days
Wipe	Lab prepared	100 sq. cm	SVOCs	EPA Method 8270	14 days
Contact/Tape	ZEFON – Biotape Surface sampler	Direct/Point	Physical Character	Optical Micro	Indefinite
Wipe	Alcohol wipes	As applicable	Petroleum Hydrocarbons	GC/MS Qualitative fingerprinting	

Following the sampling, the sample will be containerized/sealed as appropriate per the lab specifications, labeled with a sample point identification number, stored for shipment in compliance with the test method and laboratory standards, logged on a chain of custody, and will be submitted to a laboratory for analysis at the end of each day. The analytical laboratories will be instructed to note analytes that are detected below the reporting limit but above the method detection limit (i.e. a J-Flag reported quantity). Rush turnaround time will be requested to receive the laboratory results as early as possible.

4.4 Soil and Other Source Characterization Sampling

In addition to indoor environmental sampling, DPH will conduct sampling to characterize surficial soils near the Well SS-25, and at the perimeter of the affected neighborhoods. DPH will also investigate the nature of drilling fluids injected into the well. Soil sampling and other source characterization activities will be used to determine whether or not concentrations of chemical compounds and/or particulate matter measured in indoor air or on indoor surfaces are associated with the Well SS-25 gas leak.

5.0 QUALITY ASSURANCE

This section describes the analytical techniques and QA/QC procedures that will be used to provide that the data gathered during this investigation are of high quality, valid, and defensible. Data quality objectives (DQOs) are also discussed.

5.1 Project Management

DPH and its consultant Leighton are responsible for oversight of implementing this Plan. DPH and Leighton will coordinate team personnel from subcontractors and University of California Los Angeles (UCLA) to implement this protocol.

Project/Task Organization

Personnel from subcontractors and UCLA will conduct project tasks at the site and field sample/data collection services.

5.2 Data Quality Objectives

The DQOs for this project are as follows:

To collect samples of indoor air and indoor particulate matter sufficient for laboratory analysis and characterization of the residential indoor environmental conditions.

5.3 Measurement Data Acquisition

The following subsections discuss the sampling design process, analytical laboratory requirements, Field QC requirements, and data acquisition, management, and reporting.

5.3.1 Sampling Process Design

The sampling process design consists of approximately 100 individual residential units in the target area and 10 residential units in a control group located away from the target area to be used for data comparison. Data will be acquired by collecting indoor air and surface particulate samples.

5.3.2 Laboratory Requirements

Samples will be analyzed by State of California-certified laboratories using the methods discuss in Section 4. Laboratory measurement criteria are in accordance with DQOs and the individual test methods.

5.4 Field Quality Control Requirements

The principal goal of this sampling and analysis program is to obtain accurate and representative indoor environmental samples and to provide valid analytical data. To achieve these goals, QA/QC samples consisting of, trip blanks, field blanks, and replicate samples, will be collected in the field. Replicate samples will be collected at a frequency of 1 per 10 investigative samples (10 percent). A minimum of one trip blank and field blank per day will be collected for each media. Use of the data is discussed in the following sections.

5.5 Data Management

The laboratory will verify, reduce, and report data as specified in its laboratory QA plan and the laboratory statement of work for this project. Hard-copy data will be delivered along with a case narrative, table of contents, and raw data for level DQO deliverables for all data packages. Investigators will manage, reduce, and report data as described in the following sections.

5.5.1 Data Reduction

Data reduction calculations will be performed by an experienced professional knowledgeable in the meaning and use of relevant equations. Data validation reports will be summarized and included in the project report. This summary will focus on changes to the data, especially rejected data, violations of protocol, recurring problems, and data qualified as not detected because of blank contamination. A summary of reasons for changes will be included in the project report.

5.5.2 Reporting

Preliminary data results will be entered into electronic format and submitted for preliminary decision-making on the environmental status of the sites. No validation procedures or calculations will be conducted on these preliminary data.

The hard-copy laboratory reports and the updated analytical database will be used for data validation. Data validation results, analytical findings, and a comparison of data relative to the DQOs will be summarized in the body of the project report. Analytical data sheets and data validation findings will be included as an appendix to the project report.

5.5.3 Assessment and Oversight

Overall responsibility for implementing and monitoring the QA program resides with the project manager. The project manager will be responsible for reviewing the technical contents of all submittals required under this project.

5.6 Data Review, Validation, and Verification Requirements

Laboratory deliverables will be validated to assess method compliance, calibration frequency and acceptability, QC frequency and acceptability, and data usability when failures are identified. Data generated from the subject properties will be validated under USEPA Level II guidelines. Qualifiers indicating the usability of data will be attached to results as appropriate.

5.7 Assessment of Measurement Performance

For analytical results, various qualifiers pertaining to the quality of the data are attached to certain data by either the laboratories conducting the analysis or by persons conducting the data evaluation. The laboratory will flag and report estimated concentrations before reporting the data. All qualifiers will be discussed before using the chemical data for the evaluation.

5.8 Data Quality Assessment

The evaluation of the analytical data will include an assessment of precision, accuracy, representativeness, comparability, and completeness (PARCC) parameters. Reviewing particular types of QC sample results against pre-established acceptance limits assesses each of the PARCC parameters.

6.0 REPORTING

Upon completion of field activities and receipt of final laboratory analytical reports, a technical document will be prepared which at a minimum will include the following elements:

- Executive Summary,
- Site Description & Background,
- Sampling Activities and Results,
- Photographs
- Figures,
- Tables, and
- Conclusions and Recommendations

Public Health will be provided with preliminary laboratory reports as they are received from the laboratory on an ongoing basis during the field sampling process. A draft report will be submitted within one week of receipt of the final analytical reports. The report will be finalized within three (3) business days of receiving compiled comments from Public Health. Upon finalizing the report, Public Health will be provided with five hard copies, and two Compact Disc (CD) copies of the final document. The document will be signed by the appropriate State certified professionals.

7.0 REFERENCES

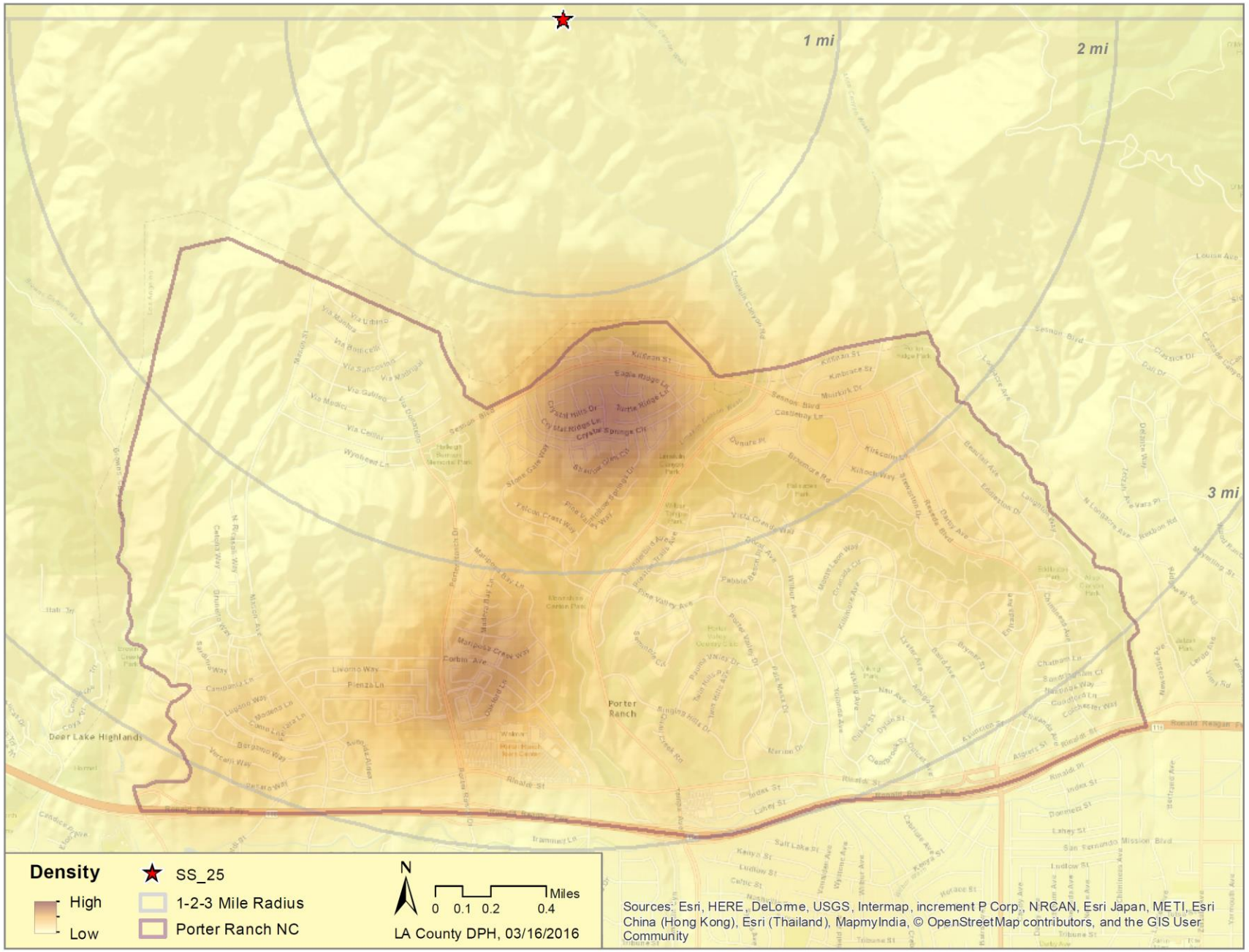
County of Los Angeles Public Health, 2016, Aliso Canyon Gas Leak, Results of Air Monitoring and Assessments of Health, Second Supplemental Report, dated February 19, 2016.

Environmental Protection Agency (EPA), 2006, Guidance on Systematic Planning Using the Data Quality Objectives Process (EPA/240/B-06/001), dated February 2006.

Office of Environmental Health Hazard Assessment (OEHHA), 2016, OEHHA Evaluation of “Criteria for Determining when Air Quality in the Porter Ranch and Surrounding Communities has Returned to Typical (Pre-SS-25 Leak) Levels”.

South Coast Air Quality Management District (SCAQMD) and California Air Resources Board (CARB), 2016, Criteria for Determining when Air Quality in the Porter Ranch and Surrounding Communities has Returned to Typical (Pre-SS-25 Leak) Levels, dated February 16, 2016.

Figure 1. Community of Porter Ranch Target Sample Area
Community Indoor Environmental Sampling Plan
Aliso Canyon Natural Gas Incident



APPENDIX

SAMPLE COLLECTION GUIDES

TO-13A (MODIFIED)

Contact your local ALS Technical Sales Representative for more information.

Polycyclic Aromatic Hydrocarbons (PAHs) in Ambient Air Using Gas Chromatography/ Mass Spectrometry

This modification of EPA Method TO-13A is used for applications involving low volume sampling of PAHs in ambient air using a combination of Polyurethane foam (PUF) and XAD resin packed into a tube. After extraction, the compounds are introduced into a gas chromatograph where they are then identified and quantified by mass spectrometry. Refer to the method for further details (http://www.epa.gov/ttn/amtic/files/ambient/airtox/to-13arr.pdf).

Equipment

1. Air sampling pump capable of sampling at 5L/min for 4-24 hours with the sampling medium in-line
2. Airflow calibrator (ex., bubble meter, Bios DryCal flow meter, etc.)
3. PUF/XAD tube – preconditioned from laboratory
   Tubes may be stored wrapped in aluminum foil at ambient temperature prior to use. After sampling, tubes must be stored cold, and must be shipped cold to the laboratory.
4. Field blank - A field blank is recommended for each sampling event. Field blanks should be subjected to the exact same handling as the samples (open, seal, and transport), except that no air is drawn through them.

Sampling Guide

Sampling Flow Rate - 1 - 5 L/min

Air Volume - Up to 7200 L for ambient and indoor air. ***Do not exceed maximum volume.***

Sample Time – Maximum of 24 hours

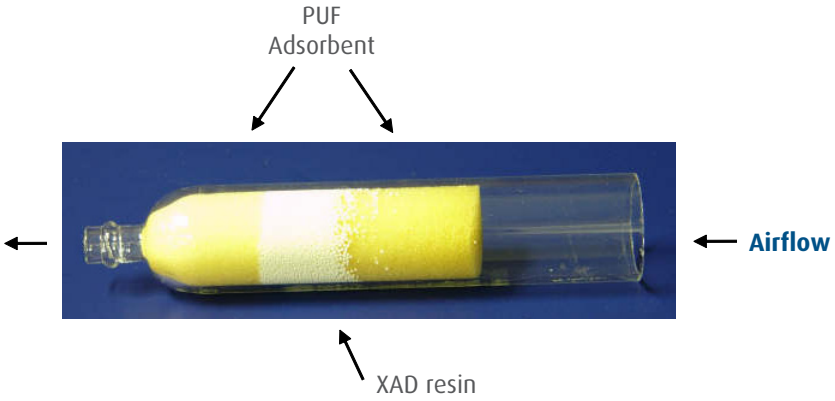
Continued from reverse side...

If Sampling Pump is Not Received Pre-calibrated:

1. Using an airflow calibrator, calibrate pump with representative media inline, following directions provided from vendor. Use the calibration tube provided from the lab. ***Do not use a sample tube.***

Sampling with a Calibrated Pump:

1. Remove the sample tubes from the shipping container.
2. Unwrap the cartridge from the aluminum foil. Do not write/scratch any additional information or put labels or tape on the tube.



1. Locate the thin end of the sampling tube. Place the thin end of the tube into the flexible tubing attached to the sampling pump.
2. Set up the sampling tube in the sampling location.
3. Turn the pump on and note the starting time and date.
4. If collecting a field blank, unwrap the field blank tube to expose it to field conditions, and then immediately rewrap the tube. Place the field blank tube back in the storage container.
5. Sample at a known flow rate for the recommended period of time, approximately 4 - 24 hours. ***Do not exceed maximum recommended volume of 7200 L.***
6. At the end of the sampling period, retrieve the sampler, turn the pump off and record the final sampling time.
7. Rewrap all samples with aluminum foil and place the tubes back in the glass jar they came in.
8. Please note all pertinent sampling information (sample identification, sample date, etc.) on the adhesive label located on the outside of the jar prior to shipping.

Storage and Shipping Instructions

1. Carefully pack sample tubes and field blank in a cooler containing frozen blue ice packs. Be sure to include all pertinent information (e.g. sample identification, sampling date, time and sample volume, etc.,) on the Chain of Custody form that is submitted with the samples.
2. Ship the cooler to the laboratory using an overnight courier service (FedEx, UPS, etc.). If unable to ship the samples back to the laboratory that day, store the samples in sealed containers at 4° C in a solvent-free refrigerator.
3. Please note that since these cartridges are reusable, any unused or broken media should be returned directly to the laboratory.

CANISTER SAMPLING

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When sampling using a canister, there are two primary modes of sampling: "GRAB" sampling and "TIME INTEGRATED" sampling. For GRAB sampling, the canister valve is simply opened and the vacuum inside the canister draws in a sample within a matter of seconds. GRAB sampling is most often used for discrete odor events, or for static concentration sample streams. TIME-INTEGRATED samples require an additional piece of laboratory calibrated equipment (flow controller or critical orifice) to be placed in-line with the canister. Flow controllers/critical orifice assemblies are equipped with fine particulate filters and are set for any user-defined duration (or flow rate) from 5 minutes up to 24 hours.

Required Items and Equipment

1L size canister with analog gauge and critical orifice assembly

- REQUIRED: 9/16" Wrench, Adjustable Crescent Wrench, and Sample Tags Supplied by ALS
- Summa or Silco canister – cleaned and certified by ALS Environmental, and leak checked prior to shipment. Canisters are available in several sizes, including 6L and 1L.
- Flow controller (a.k.a. "regulator") – Used to collect a time-integrated indoor air or ambient air sample. Flow controllers are calibrated by the laboratory for your project specific requirements; please do not adjust any of the settings or knobs.
- Critical Orifice Assembly (COA) – Used to collect a time-integrated soil gas, sub-slab, SVE system, or other vapor sample. COAs are precisely calibrated by the laboratory for your project specific requirements; please do not disassemble any parts on the assembly.
- Analog gauge – Gauge on Swagelok ¼" Tee fitting, used to monitor pressure during sampling. Note that these gauges are for general reference purposes only, and canister vacuum is checked prior to shipping and upon receipt at the laboratory.

CANISTER SAMPLING INSTRUCTIONS

Procedure

1. Ensure that the canister valve is fully closed (the canister valve should be turned completely clockwise).
2. Using a 9/16" wrench, remove the brass cap from the valve on the top of the summa canister.
3. If desired, attach the analog gauge (on a Swagelok Tee) to the valve on the top of the canister. Tighten down with your fingers first, then tighten gently with 9/16" wrench. Stabilize the backside of the valve with an adjustable crescent wrench.
4. To check the canister initial pressure, re-attach the brass cap to the top of the analog gauge. Tighten down with your fingers first, then tighten gently with 9/16" wrench.
5. Open the can approximately 1 ¼ turns, and note the initial pressure reading on the chain of custody. Please note that if the gauge does not equilibrate within 30 seconds or appears to be losing vacuum, the canister is leaking due to a loose fitting. Close the canister valve immediately and tighten the fittings.
6. If collecting a GRAB sample, disconnect the brass cap from the top of the canister and open the canister valve, turning the valve counterclockwise until there is no resistance. Then turn back clockwise slightly until resistance is detected. You will hear a hissing noise as the vacuum dissipates and draws air in. Then skip to step number 10.
7. If collecting a TIME INTEGRATED sample, disconnect the brass cap and attach to the analog gauge on the top of the caniser. Tighten the fitting with your fingers first, then tighten gently using 9/16" wrench.
8. Perform a flow regulator SHUT IN test as shown on the next page.

Step 3

9. To open the canister valve, turn the valve counterclockwise until there is no resistance. This is approximately 1 ¼ turns. Then turn back clockwise slightly until resistance is detected. Since the flow controller restricts the air flow, you will NOT hear a hissing noise as the vacuum dissipates and draws air in.
10. At the end of the sampling period, close the canister valve by turning the knob clockwise. Do not tighten with a wrench.
11. Remove all attached equipment from the canister and wrap in bubble wrap for shipment.
12. Replace the brass cap on the canister valve. Tighten using a 9/16" wrench.
13. Label the sample with the tag provided, and attach the tag to the canister with the provided plastic ties.
14. Complete a chain of custody form. Please note the canister barcode ID number on the COC. For time-integrated sampling, please also note the flow controller or critical orifice assembly identification number with the corresponding canister.
15. Place the chain of custody form, the bubble-wrapped flow controller, and the canister back into the original boxes in which they were shipped to you.

CONDUCTING A FLOW REGULATOR "SHUT IN" TEST

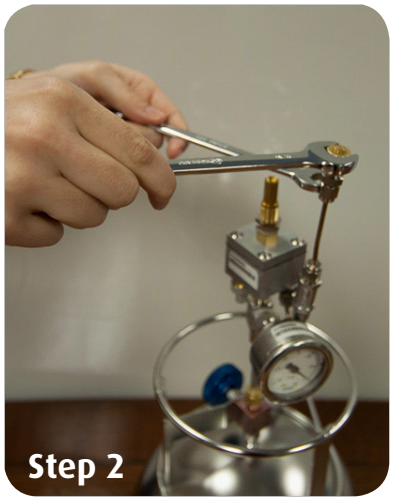
When utilizing a flow controller or critical orifice assembly, it is recommended that a "SHUT-IN" test be performed. The purpose of a SHUT IN test is to create a closed system between the canister, gauge, and flow regulator, where any loose connections in the system can be easily identified prior to sample collection.

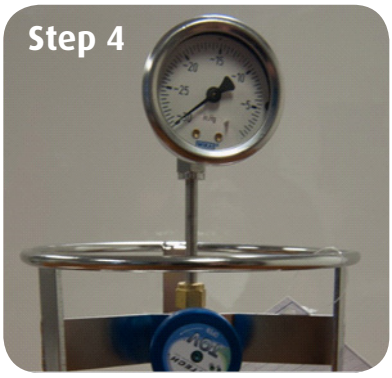
If you are only collecting a "GRAB" sample, please disregard this set of instructions and proceed to the "TROUBLESHOOTING GUIDE" on the backside of this document if necessary.

Procedure

1. Perform steps 1-7 according to the Canister Sampling Instructions.
2. After the setup has been assembled, attach the brass cap to the end of the flow regulator. Please note that for critical orifice assemblies, the nut and pink rubber ferrule may need to be removed prior to attachment. Tighten the fitting with your fingers first, then tighten gently using 9/16" wrench.
3. Open the canister valve counterclockwise approximately 1 ¼ turns, and leave it open for roughly 10-15 seconds. After the time has elapsed, turn the valve clockwise to close.

   Please note that the canister valve is being left open for 10-15 seconds in order to allow the closed system to equilibrate. Since there is considered to be some "dead volume" in the tubing between in the canister valve and the brass cap, by letting the canister equilibrate it avoids misinterpreting the slight change in vacuum as a loose fitting.
4. After the valve is closed, study the vacuum gauge for approximately three to five minutes. If the vacuum gauge shows more than approximately 1.0 in Hg, change in pressure per minute, then there is a loose fitting. Re-tighten all fittings, and continue to wait to see if the canister retains its vacuum. If the canister retains pressure, it is considered to be "leak-tight".
5. Ensure valve is closed, remove brass cap, and continue with steps 9-15 according to the Canister Sampling Instructions.





CANISTER SAMPLING TROUBLESHOOTING & FREQUENTLY ASKED QUESTIONS

1. What do I do with the digital gauge provided?

If a digital gauge has been provided, it is to be used for checking the initial pressure ONLY. Due to the qualitative nature of the analog gauges, a digital gauge has been provided to assist in accurately establishing the initial vacuum of a canister, however if it is used after sampling occurs, then there is a possibility of sample cross-contamination.

2. The valve on my canister is twisting around in circles. Is this normal?

If you have a canister with a blue valve, then yes. These canisters contain special ferrules that allow the valve to make full revolutions with no effect to the canister vacuum. The fittings are not loose, so please do not tighten. If tightened, there is a chance that the now compressed ferrule could cause the valve stem to leak, corrupting the sample.

3. The analog gauge does not move when I'm checking the canister vacuum or when the canister has begun sampling. What could be the problem?

Due to jostling during shipping, as well as "wear and tear" from previous field work, the analog gauge needle may be stuck. Either disconnect the gauge from the canister and tap the gauge with the back of your hand prior to re-assembly, or try another gauge.

4. How do I connect tubing to the regulator?

If you have a flow controller (small and boxy in shape, used for ambient applications), then the tubing is connected to the sample inlet, located at the open end of the "U" shaped 1/8'' metal tubing. A nut and ferrule need to have been requested separately.

If you have a critical orifice assembly ("L" shaped metal tubing, used for source applications), then unscrew the nut located at the long end of the orifice. Inside should be a pink ferrule and small ceramic frit. Place the frit back inside of the COA, and attach the tubing according to the paragraph below.

First place provided nut over the tubing, then slide the pink ferrule on to the tubing with the wider side of the ferrule closest to the nut, and the narrow side closest to the end of the tubing. Attach the nut to the regulator threads and twist until finger tight, and then tighten until snug using a 9/16'' wrench.

5. The canister seems to be filling faster or slower than the specified flow regulator duration I requested. What should I do?

As part of the ALS Laboratory QA/QC protocol, each flow regulator undergoes both a pre-calibration prior to shipping the regulator to the client, as well as a post-calibration after the regulator has been returned by the client. If a regulator does not pass the pre-calibration phase, then the regulator undergoes maintenance and re-cleaning, and if it does not pass the post-calibration after receipt, then the laboratory Project Manager is required to notify the client.

1. For field troubleshooting: Close the canister valve, and try tapping the gauge with your finger or back of your hand and make sure that it stays steady. Due to vigorous conditions in the field, gauges sometimes require manual "re-calibration".

*If your canister is filling **slower** than expected:*

Is there high humidity, or visible water vapor in the inlet tubing? Is there visible particulate matter, or are you taking a soil gas sample? Each flow regulator is outfitted with an in-line filter, which is designed to keep particulate matter and water vapor out of the canister. If either applies, there is a potential that the filter is clogging the regulator inlet.

If this does not apply, try outfitting the canister with a different gauge and trying again.

*If your canister is filling **faster** than expected:*

Did you perform a shut-in test prior to sampling? If not, then it is likely there is a leak in the sampling train, which is causing the expedited sample time.

- If this is an ambient air sample, close the valve to terminate sampling, perform a shut-in test, and then begin sampling again; since the ambient air is being collected as sample, there should not be any contamination issues from the leak.
- If this is a "source" level sample (i.e. soil gas, SVE system, landfill gas, using tubing to connect to a source, etc.), and you have not performed a shut-in test, stop your sample immediately. Perform a shut-in test on the sampling train to confirm leak, and if there is a leak (i.e. the canister has pulled ambient air), please return the contaminated can back to the laboratory and use a separate canister for sampling.

2. If none of these scenarios apply, please inquire with your ALS Project Manager for further troubleshooting.

6. How tight should the canister valve be after sampling completion? What about the brass cap?

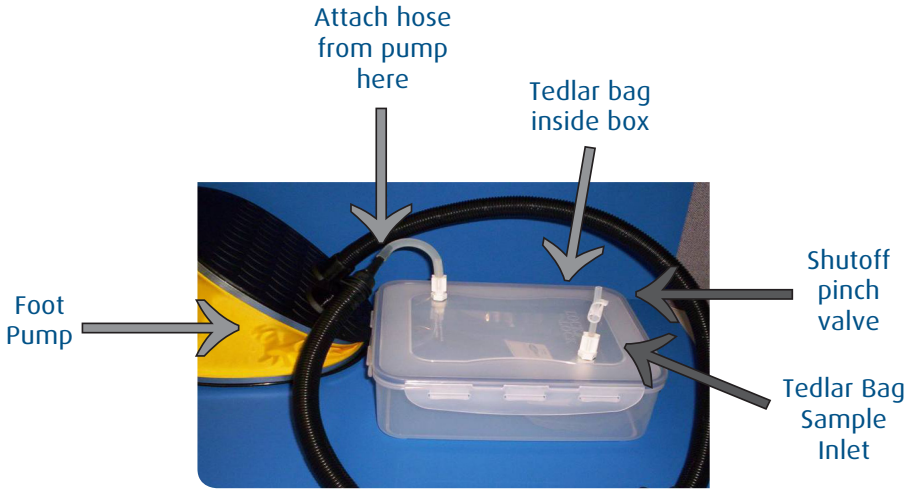
After sampling elapses, the valve only needs to be finger tight. Please do not ever use a wrench to tighten. For the brass cap, please twist the cap on using your finger, until it is "finger-tight". From there, use a 9/16'' wrench to ensure it is snug – but DO NOT OVERTIGHTEN.

LUNG SAMPLING INSTRUCTIONS

Contact your local ALS Technical Sales Representative for more information.

A lung sampler facilitates the collection of an air sample in a Tedlar bag without passing air directly through a pump, thereby reducing the possibility of contaminating the pump, the sample and future samples. It does this by creating a vacuum inside the sampler, which forces the Tedlar bag to expand and draw in an air sample through the sampler inlet.

1. Record the sample identification, sampling date, and time on the label of the Tedlar bag.
2. Ensure that the shutoff pinch valve on the sampler inlet is closed. Open the sampler and place the Tedlar bag on the bottom of the container with the valve pointing upwards. Using the small piece of the silicon tubing, connect the valve of the bag to the sampler inlet tubing. Open the valve on the bag by turning the valve counter clockwise approximately 1 ¼ times.
3. Close and seal the container.
4. Attach the foot pump to the vacuum port via the Teflon tubing. Do not connect the foot pump to the sampler inlet.
5. To begin sampling, open the shutoff pinch valve to the inlet.
6. Pump the foot pump one to two times.
7. The bag will begin to fill as a vacuum is created inside the sampler.
8. Once the bag is 1/2 to 2/3 full, pinch closed the inlet valve on the outside of the container. Open the sampler and immediately close the valve on the bag.



LUNG SAMPLER SETUP

Scan the QR code or visit
WWW.ALSGLOBAL.COM

Continued from reverse side...

Correctly Inflated Tedlar Bag

Additional Information

Reduced sulfur compounds , such as hydrogen sulfide, submitted for analysis by ASTM D5504-08 have a 24-hour holding time. Volatile organic compounds to be analyzed by EPA TO-15 have a holding time of 72 hours.

Tedlar bags should be packed in a rigid container, like a box or cooler, free from any loose items that could rupture the bags, and shipped by overnight courier to the laboratory. They are not required to be shipped cold.

Esta página en Español

ELEMENTS by ICP (Nitric/Perchloric Acid Ashing) 7300

MW: Table 1 CAS: Table 2 RTECS: Table 2

METHOD: 7300, Issue 3 **EVALUATION: PARTIAL** **Issue 1: 15 August 1990**
Issue 3: 15 March 2003

OSHA: Table 2
NIOSH: Table 2
ACGIH: Table 2

PROPERTIES: Table 1

ELEMENTS:

aluminum*	calcium	lanthanum	nickel	strontium	tungsten*
antimony*	chromium*	lithium*	potassium	tellurium	vanadium*
arsenic	cobalt*	magnesium	phosphorus	tin	yttrium
barium	copper	manganese*	selenium	thallium	zinc
beryllium*	iron	molybdenum*	silver	titanium	zirconium*
cadmium	lead*				

*Some compounds of these elements require special sample treatment.

SAMPLING

SAMPLER: FILTER (0.8-µm, cellulose ester membrane, or 5.0-µm, polyvinyl chloride membrane)

FLOWRATE: 1 to 4 L/min

VOL-MIN: Table 1
-MAX: Table 1

SHIPMENT: routine

SAMPLE STABILITY: stable

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: not determined

BIAS: not determined

OVERALL PRECISION ($\hat{S}_{rT}$): not determined

ACCURACY: not determined

MEASUREMENT

TECHNIQUE: INDUCTIVELY COUPLED ARGON PLASMA, ATOMIC EMISSION SPECTROSCOPY (ICP-AES)

ANALYTE: elements above

ASHING REAGENTS: conc. HNO_3/ conc. $HClO_4$ (4:1), 5 mL; 2mL increments added as needed

CONDITIONS: room temperature, 30 min; 150 °C to near dryness

FINAL SOLUTION: 4% HNO_3, 1% $HClO_4$, 25 mL

WAVELENGTH: depends upon element; Table 3

BACKGROUND CORRECTION: spectral wavelength shift

CALIBRATION: elements in 4% HNO_3, 1% $HClO_4$

RANGE: varies with element [1]

ESTIMATED LOD: Tables 3 and 4

PRECISION ($\bar{S}$): Tables 3 and 4

APPLICABILITY: The working range of this method is 0.005 to 2.0 mg/m^3 for each element in a 500-L air sample. This is simultaneous elemental analysis, not compound specific. Verify that the types of compounds in the samples are soluble with the ashing procedure selected.

INTERFERENCES: Spectral interferences are the primary interferences encountered in ICP-AES analysis. These are minimized by judicious wavelength selection, interelement correction factors and background correction [1-4].

OTHER METHODS: This issue updates issues 1 and 2 of Method 7300, which replaced P&CAM 351 [3] for trace elements. Flame atomic absorption spectroscopy (e.g., Methods 70XX) is an alternate analytical technique for many of these elements. Graphite furnace AAS (e.g., 7102 for Be, 7105 for Pb) is more sensitive.

REAGENTS:

1. Nitric acid (HNO_3), conc., ultra pure.
2. Perchloric acid ($HClO_4$), conc., ultra pure.*
3. Ashing acid: 4:1 (v/v) HNO_3:$HClO_4$. Mix 4 volumes conc. HNO_3 with 1 volume conc. $HClO_4$.
4. Calibration stock solutions, 1000 µg/mL. Commercially available, or prepared per instrument manufacturer's recommendation (see step 12).
5. Dilution acid, 4% HNO_3, 1% $HClO_4$. Add 50 mL ashing acid to 600 mL water; dilute to 1 L.
6. Argon.
7. Distilled,deionized water.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: cellulose ester membrane filter, 0.8-µm pore size; or polyvinyl chloride membrane, 5.0-µm pore size; 37-mm diameter, in cassette filter holder.
2. Personal sampling pump, 1 to 4 L/min, with flexible connecting tubing.
3. Inductively coupled plasma-atomic emission spectrometer, equipped as specified by the manufacturer for analysis of elements of interest.
4. Regulator, two-stage, for argon.
5. Beakers, Phillips, 125-mL, or Griffin, 50-mL, with watchglass covers.**
6. Volumetric flasks, 10-, 25-,100-mL., and 1-L**
7. Assorted volumetric pipets as needed.**
8. Hotplate, surface temperature 150 °C.

** Clean all glassware with conc. nitric acid and rinse thoroughly in distilled water before use.

SPECIAL PRECAUTIONS: All perchloric acid digestions are required to be done in a perchloric acid hood. When working with concentrated acids, wear protective clothing and gloves.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Sample at an accurately known flow rate between 1 and 4 L/min for a total sample size of 200 to 2000 L (see Table 1) for TWA measurements. Do not exceed a filter loading of approximately 2 mg total dust.

SAMPLE PREPARATION:

3. Open the cassette filter holders and transfer the samples and blanks to clean beakers.
4. Add 5 mL ashing acid. Cover with a watchglass. Let stand 30 min at room temperature.
   NOTE: Start a reagent blank at this step.
5. Heat on hotplate (120 °C) until ca. 0.5 mL remains.
   NOTE 1: Recovery of lead from some paint matrices may require other digestion techniques. See Method 7082 (Lead by Flame AAS) for an alternative hotplate digestion procedure or Method 7302 for a microwave digestion procedure.
   NOTE 2: Some species of Al, Be, Co, Cr, Li, Mn, Mo, V, and Zr will not be completely solubilized by this procedure. Alternative solubilization techniques for most of these elements can be found elsewhere [5-10]. For example, aqua regia may be needed for Mn [6,12].
6. Add 2 mL ashing acid and repeat step 5. Repeat this step until the solution is clear.
7. Remove watchglass and rinse into the beaker with distilled water.
8. Increase the temperature to 150 °C and take the sample to near dryness (ca. 0.5 mL).
9. Dissolve the residue in 2 to 3 mL dilution acid.
10. Transfer the solutions quantitatively to 25-mL volumetric flasks.
11. Dilute to volume with dilution acid.
    NOTE: If more sensitivity is required, the final sample volume may be held to 10 mL.

CALIBRATION AND QUALITY CONTROL:

12. Calibrate the spectrometer according to the manufacturers recommendations.
    NOTE: Typically, an acid blank and 1.0 µg/mL multielement working standards are used. The following multielement combinations are chemically compatible in 4% HNO_3/1% $HClO_4$:
    a. Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, La, In, Na
    b. Ag, K, Li, Mg, Mn, Ni, P, Pb, Se, Sr, Tl, V, Y, Zn, Sc
    c. Mo, Sb, Sn, Te, Ti, W, Zr
    d. Acid blank
13. Analyze a standard for every ten samples.
14. Check recoveries with at least two spiked blank filters per ten samples.

MEASUREMENT:

15. Set spectrometer to conditions specified by manufacturer.
16. Analyze standards and samples.
    NOTE: If the values for the samples are above the range of the standards, dilute the solutions with dilution acid, reanalyze and apply the appropriate dilution factor in the calculations.

CALCULATIONS:

17. Obtain the solution concentrations for the sample, C_s (µg/mL), and the average media blank, C_b (µg/mL), from the instrument.
18. Using the solution volumes of sample, V_s (mL), and media blank, V_b (mL), calculate the concentration, C (mg/m^3), of each element in the air volume sampled, V (L):

$$C = \frac{CsVs - CbVb}{V}, mg/m^3$$

NOTE: µg/L ≡ mg/m^3

EVALUATION OF METHOD:

Issues 1 and 2
Method, 7300 was originally evaluated in 1981 [2,3]. The precision and recovery data were determined at 2.5 and 1000 µg of each element per sample on spiked filters. The measurements used for the method evaluation in Issues 1 and 2 were determined with a Jarrell-Ash Model 1160 Inductively Coupled Plasma Spectrometer operated according to manufacturer's instructions.

Issue 3
In this update of NIOSH Method 7300, the precision and recovery data were determined at approximately 3x and 10x the instrumental detection limits on commercially prepared spiked filters [12] using 25.0 mL as the final sample volume. Tables 3 and 4 list the precision and recovery data, instrumental detection limits, and analytical wavelengths for mixed cellulose ester (MCE) and polyvinyl chloride (PVC) filters. PVC Filters which can be used for total dust measurements and then digested for metals measurements were tested and found to give good results. The values in Tables 3 and 4 were determined with a Spectro Analytical Instruments Model End On Plasma (EOP)(axial) operated according to manufacturer's instructions.

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[12] Certification Inorganic Ventures for spikes.

METHOD REVISED BY:

Mark Millson and Ronnee Andrews, NIOSH/DART.

Method originally written by Mark Millson, NIOSH/DART, and R. DeLon Hull, Ph.D., NIOSH/DSHEFS, James B. Perkins, David L. Wheeler, and Keith Nicholson, DataChem Labortories, Salt Lake City, UT.

Surface Particulate Sampling Plan

Surface Particulate Sampling Locations

Surface particulate samples will be collected from a single and randomly selected surface in each home. Potential sampling locations may include hard flooring, counter tops and/or window sills.

Only smooth, impervious surfaces, such as glass, enameled or laminated surfaces, coated surfaces, and vinyl or ceramic floors should be sampled using wipe and contact sampling protocols. Wipe sampling protocols are not applicable for surfaces with porous or non-impervious media (e.g., concrete, wood, asphalt, plasterboard).

Wipe Sampling Protocol

Wipe samples should be collected over a 100 cm^2 surface area using laboratory provided wipes and disposable 100 cm^2 templates. One template will be needed for each sample. Dispose of template after one use. The wipe should be swiped horizontally back and forth, top to bottom and vertically up and down, left to right to collect each sample.

Samples should be placed in pre-cleaned 4 ounce glass with Teflon-lined caps. Seal the jar with a Teflon-lined cap, label the jar, and store the jar on blue ice (at 4°C) until all the samples are collected.

Wipe Sampling Procedures

1. Wear a pair of clean disposable nitrile gloves.

2. Place the disposable 100 cm^2 template over the surface area to be sampled, and secure the outside edges with blue masking tape.

3. Remove the laboratory prepared wipe (or gauze pad) from its container and unfold it.

4. Re-fold the wipe into fourths, and wipe the surface area with firm pressure. Use an overlapping “S” wiping pattern described above to cover the entire surface area with five (5) horizontal strokes.

5. Fold the exposed side of the wipe in, and wipe the same area using five (5) vertical “S” strokes.

6. Fold the wipe once more to reveal an unexposed surface, and wipe the surface a third time as described in Item 4.

7. After sampling, fold the wipe, exposed side in, and place the wipe sample in a clean sample jar. Seal securely. Label the sample container clearly.

8. A new, disposable template should be used for each sample location.

9. Replace the outer pair of gloves for each sample. Discard gloves. Used gloves can be disposed of in a durable, sealable bag, such as a ZipLock bag.

Contact Sampling Protocol

Contact samples must be collected on a single sample point, adjacent to the location where the wipe sample was obtained.

Contact Sampling Procedures

1. Remove the Biotape slide from the provided slide cartridge.

2. Document the sample location on the slide, label.

3. Peel off protective liner from slide to expose adhesive and dispose of it - do not replace it after sampling.

4. Place slide with the adhesive side down on the surface to be sampled (Use the centering line to orient the adhesive over the exact sample location desired).

5. Gently press down to ensure contact is made. Do not press down hard. The adhesive is very tacky and only needs to be pressed gently.

6. Carefully remove slide from the surface and place back into slide cartridge.

7. Replace the outer pair of gloves for each sample. Discard gloves. Used gloves can be disposed of in a durable, sealable bag, such as a ZipLock bag.