

California Environmental Protection Agency



COMMUNITY AIR MONITORING BRANCH

**STANDARD OPERATING PROCEDURE
FOR
In-Situ Measurements of Atmospheric Volatile Organic
Compounds Using Thermal Desorption Gas Chromatography**

CAMB SOP 259
First Edition

**Monitoring and Laboratory Division
May 2019**

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California Environmental Protection Agency



Approval of Standard Operating Procedure

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SOP: CAMB SOP 259, First Edition
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Acronyms

AMTS	Advanced Monitoring Techniques Section
CAMB	Community Air Monitoring Branch
CARB	California Air Resources Board
CTD	Collection and Thermal Desorption
FID	Flame Ionization Detection
GC	Gas Chromatography
He	Helium
ID	Inside Diameter
LOQ	Limit of Quantification
MDL	Method Detection Limit
MS	Mass Spectrometry
N ₂	Nitrogen
NIST	National Institute of Standards and Technology
NLB	Northern Laboratory Branch
nm	Nanometer
ppb	Parts Per Billion
QCM	Quality Control Manual
RH	Relative Humidity
SOP	Standard Operating Procedure
TD	Thermal Desorption
UHP	Ultra-High Purity
VOC	Volatile Organic Compound

1.0 General Information

1.1 Introduction

This Standard Operating Procedure (SOP) describes procedures used by the California Air Resources Board (CARB) Community Air Monitoring Branch (CAMB) to operate a thermal desorption gas chromatograph-flame ionization detection (TD-GC-FID) instrument to measure volatile organic compounds in ambient air. This procedure is designed to supplement the manufacturer's manual by describing hardware or operating procedures as implemented by CAMB. It is not the intent of this SOP to duplicate or replace the manufacturer's instruction manual.

1.2 Principle of Operation

- 1.2.1 This instrument is capable of automated, unattended and around-the-clock measurements of volatile organic compounds by integrating sampling, pre-concentration, thermal desorption and GC-FID methods. This method is capable of measuring hourly variations of VOC concentrations.
- 1.2.2 This instrument has two modes of operation: the sampling mode and the analysis mode. These two modes can operate concurrently. For example, an air sample can be collected while the previous sample is being analyzed. This alternation of modes is achieved by interfacing the sampling and analysis systems through a three-way valve. This valve connects these two systems during the analysis mode and isolates these two systems during the sampling mode. Appendices A and B provide more information about the flow path of analysis and sampling systems.
- 1.2.3 During sampling, ambient air is first drawn through a water removal trap to reduce the dew point to -30°C ; then through an adsorbent tube to collect and concentrate VOCs at -30°C .
- 1.2.4 The analysis mode begins after the completion of sampling. During the analysis mode, VOCs collected by the adsorbent tube are thermally desorbed by heating the adsorbent tube to 280°C and injected into the GC-FID for analysis. Following the injection, the sampling system is isolated from the analysis system and prepared for the next sample collection.
- 1.2.5 The chromatographic separation of VOCs is performed through a two column system connected using a dean switch to achieve an optimal separation of both light and heavy VOCs. VOCs eluted from the GC columns are quantified by FID.

2.0 Definitions

- 2.0 BLANK – An aliquot of high purity nitrogen gas or zero air which is analyzed and used to monitor the analytical systems for interferences and contamination. The collection and analysis method is the same as the method used for atmospheric samples.

- 2.1 BREAKTHROUGH – Determination if any amount of sample was not retained in the cold trap during sampling due to compound structure and cold trap contents.
- 2.2 CALIBRATION CURVE – The calibration curve consists of at least five concentrations of a calibration standard that span the monitoring range of interest to determine instrument sensitivity and the linearity of GC-FID response for the target compounds.
- 2.3 CARRYOVER – Contamination from an adjacent sample causing false or inaccurate results in the subsequent sample(s).
- 2.4 INTERFERENCE – Discrete artifacts or elevated baselines from the sampling and analytical systems that may cause misinterpretation of the chromatographic data. Other interferences are matrix effects which may cause the target compound to be suppressed or enhanced giving lower or higher than expected results.
- 2.5 METHOD DETECTION LIMIT – the minimum concentration of a substance that can be measured by a single measurement and reported with 99 percent confidence that the analyte concentration is greater than zero and statistically different from a blank. It is determined from replicate analyses of a sample in a given matrix containing the analyte and sampling media. The procedure used to determine the MDL is documented in the NLB's Laboratory QCM.
- 2.6 LIMIT OF QUANTITATION - The minimum concentration or amount of an analyte that a method can measure with a specified degree of confidence. The LOQ is equal to five times the standard deviation of the replicate analyses from the MDL determination/verification. LOQ is analyte and instrument specific.
- 2.7 REPORTING LIMIT – Equivalent to or greater than the lowest level of the calibration curve. Detections below the reporting limit are typically reported as "<RL" unless otherwise requested by the client.

3.0 Interferences and Limitations

- 3.1 All target compounds are primarily identified by their retention time. Compounds, not included the target compound list, having similar GC retention times may co-elute with target compounds. This co-elution can lead to misidentification or inaccurate quantitation of target compounds. Canister samples collected at the same location periodically and analyzed by GC-mass spectrometry can be used as a reference to check whether co-elution is present and substantial. In addition, the peak shape of analytes and their time series and diurnal patterns are also checked. The target compounds will be flagged if co-elution with other analytes is confirmed.
- 3.2 The analytical system may become contaminated when samples containing high compound concentrations are analyzed. If there is suspected carryover

from a high concentration sample, additional blanks should be analyzed to clean the system.

- 3.3 High boiling point compounds trapped on the column may cause baseline shifting, or the appearance of broad, extraneous “ghost” peaks. The column should be baked out to remove these contaminants prior to analyzing samples. The bake-out temperature must not exceed the column’s maximum operating temperature. The maximum operating temperature is labeled outside the column packing box or can be looked up online using the column information which is attached to the column.

4.0 Personnel Qualifications and Training

Prior to operating this instrument, new personnel must be trained by staff with expert knowledge of this instrument. Personnel must be trained to understand the program’s requirements and this SOP. Personnel will also be trained on how to safely and properly operate the equipment needed to perform the method and the quality assurance components. Personnel should provide an initial demonstration of capability prior to performing this method on real-world samples (i.e. data for record).

5.0 Safety Requirements

- 5.1 All personnel must follow the general health and safety requirements found in MLD’s Chemical Hygiene Plans.
- 5.2 This method uses high pressure gases. Follow safe handling practices regarding compressed gases when moving and installing the cylinders. Use suitable equipment and protective devices, such as carts. Refer to Section III in the MLD Chemical Hygiene Plan for safe handling practices.
- 5.3 The standards used for calibration of this method contain toxic compounds. Precautions should be taken to limit the potential for inhalation of these compounds.
- 5.4 The GC and FID have heated zones which may cause burns. The trap is both heated and cooled. Avoid contact with these zones and devices when in operation and make certain they are de-energized or at ambient temperature prior to servicing by checking temperature gauges on instruments.

6.0 Equipment and Supplies

- 6.1 Summa polished stainless steel canisters for standards and blanks.
- 6.2 A Markes Air Server and Kori-Unity system or an equivalent sampling collection and thermal desorption (CTD) system. This system should be capable of automated water vapor control, preconcentration, thermal desorption and injection of VOCs into GC for analysis. In addition, this system should be equipped with at least three sampling ports for atmospheric

samples, standards, and blanks.

- 6.3 GC-FID for chromatographic separation and quantification of ambient VOC samples. This GC-FID system is equipped with two analytical columns (TG-1 and PLOT columns or equivalent columns) and one restrictor column and two FID detectors. All three columns are connected via a flow splitter. The TG-1 column connects the injection port to the flow splitter. The PLOT column and the restrictor column connect the flow splitter to respective FIDs. The eluted analytes from the TG-1 column is introduced into the PLOT column and the restrict column sequentially by directing the TG-1 column flow through the flow splitter. The GC flow system is further described in Appendix A.
- 6.4 Data stations, such as Thermo Scientific Chromeleon and Markes Instrument control, for controlling the GC, FID, the flow splitter and the automated sampling system.
- 6.5 TG1-MS column 60 m x 0.25 mm x 1.0 μm or equivalent.
- 6.6 HP-AL/S PLOT column 50 m x 0.32 mm x 8.0 μm or equivalent.
- 6.7 Restrictor column 0.75 m x 0.1 mm or equivalent.
- 6.8 Markes sorbent focusing trap U-T20PAM-2S or equivalent.
- 6.9 A Parker Balston hydrogen generator (H2PEM-165) or equivalent.
- 6.10 An API Model 701 Zero Air generator or equivalent.
- 6.11 A Thermo Scientific GLD pro leak detector.

7.0 Reagents and Gases

- 7.1 Nitrogen UHP, 99.999%.
- 7.2 Helium UHP, 99.999%.
- 7.3 Hydrogen UHP, 99.999%.
- 7.4 Zero Air UHP, 99.999%.
- 7.5 Gas calibration standards and control.

8.1 Field Measurements

- 8.1 Instrument Setup
 - 8.1.1 Install both the GC-FID and the sampling system on a flat and stable bench top inside a temperature controlled room or shelter (15°C to 35°C). The shelter is recommended to maintain lower than 26°C for measurements with one-hour sampling frequency.
 - 8.1.2 Connect all gas lines (nitrogen, helium, zero air, and hydrogen) to their respective ports located on the rear panel of the instruments. All of

these ports are clearly labeled.

- 8.1.3 Connect all instrument control and data acquisition cables to their corresponding ports on the instruments and computer.
 - 8.1.4 Install the GC columns.
 - 8.1.5 Connect all power cords.
 - 8.1.6 Set the pressure of the pressure regulators (60~100 psi) of nitrogen and helium cylinders, the output pressure (60 psi) of the hydrogen generator, the output pressure (~45 psi) of the zero air generator and the external pressure regulators (nitrogen and helium, 45 to 55 psi, typically at ~50psi) for the sampling system.
 - 8.1.7 Connect the sampling pump to the CTD system.
 - 8.1.8 Turn on the GC-FID and the CTD system, including the sampling pump, by flipping their respective power switches located on their rear/front panels.
 - 8.1.9 Turn on the computer and make sure that the instrument control and data acquisition stations communicate with the instruments.
 - 8.1.10 Load the instrument methods for both GC-FID and the sampling system. Methods are provided in Appendix C. Methods are named clearly for sampling, analysis or data processing.
 - 8.1.11 Conduct a leak check for both GC and the CTD system. The GLD leak detector is used to perform leak check at the connections where the carrier gas passes through. Please see 8.2.5 for the leak check for sampling inlet.
- 8.2 Sampling Inlet
- 8.2.1 One of three sampling ports of the Markes Air Server is used for sampling atmospheric air with two other ports for sampling zero air/UHP N₂ for blanks and standards for the calibration and one-point check).
 - 8.2.2 The sampling inlet is made of a section of stainless steel tubing with 1/8" outside diameter. The residence time of atmospheric air inside the sampling line should be equal to or less than 20 seconds.
 - 8.2.3 A Teflon filter or stainless steel filter (5 micron pore size or less) is placed upstream of the sampling port for removal of atmospheric particles.
 - 8.2.4 The sampling inlet should be located 2 to 15 meters above the ground.
 - 8.2.5 The sampling inlet requires leak checks. The flowrate at the sampling port and the sampling inlet outside the shelter where the auto-GC is located should be measured. The difference between the flowrate at two points should be less than one percent.

8.3 Blanks and VOC standards

- 8.3.1 One of the sampling ports of the CTD system is connected to a canister filled with UHP N₂ or zero air. The UHP N₂ (or zero air) is used for collecting blanks.
- 8.3.2 One of the sampling ports of the sampling system is connected to a canister filled with VOC standards diluted to a typical ambient concentration in the monitoring site. This sampling port is used to conduct one-point standard check and construct the calibration curve.

8.4 Ambient Sampling and Chemical Analysis

- 8.4.1 A sequence template is created to enable automated sampling and instrument analysis. The sequence consists of three types of samples: atmospheric sample, blank, and one-point check standard. The three types of samples are grouped sequentially as 22 ambient samples (or fewer), one blank, and one-point check standard. A sequence for field measurements can then be created using this template by repeating the template sequence.
- 8.4.2 Two separate sequences are needed; one for the analysis system and another for the sampling system. The number of runs in both sequences should be same. The sample type should match the sampling port.
- 8.4.3 This GC-FID makes hourly measurements. The duration of each sample is composed of 30-min sampling (sampling duration can be adjusted based on needs of measurements. See 8.4.4), 52-min GC analysis time, and 5-min GC oven cool-down time. Sampling and analysis can be conducted concurrently. Detailed description of the modes of operation is provided in Appendix B.
- 8.4.4 Sampling time is determined by the objective of the measurements, the breakthrough volume, ambient VOC concentrations, and sampling flow rate. Typically, the breakthrough volume is around 1-1.5 L of air. The actual breakthrough volume for each adsorbent trap can be determined by sampling different volume of VOC calibration standards as needed.
- 8.4.5 The sampling and chemical analysis of ambient samples is automated once the sequence is started.

8.5 Data analysis

- 8.5.1 A data processing method is created for the target compounds.
- 8.5.2 The identification of compounds is primarily based on the retention time determined using the authentic standards. The retention time window is checked daily by running the one-point check standard.
- 8.5.3 The integrated peak area is directly converted to atmospheric concentrations in a user defined unit, such as ppbv or $\mu\text{g}/\text{m}^3$, using the

response factor of each compound determined through the calibration curve using authentic standards. The calculations are performed by the data acquisition and processing station.

- 8.5.4 Compounds where the authentic standard are unavailable can also be identified and quantified. The identification can use the retention time indices reported in the literature using the similar analytical method and confirmed by GC/MS analysis of the canister samples collected at the same sampling site. The ambient concentration of these compounds can be determined by applying the response factor of other compounds where authentic standards are available, given that the FID has a similar response factor per carbon mass for hydrocarbons. The addition of the compounds to the original compound list requires approval of CAMB management based on the added value of additional analytes to the monitoring purpose and the staff time needed to analyze these raw data.
- 8.5.5 The time series of target compounds in the one-point check standard are used to check the overall instrument performance. Chromatograms need to be reviewed if unexpected spikes in concentrations and/or anomalies are observed (please see 9.0 Quality Control).

8.6 Data Reporting

All processed data are automatically compiled and reported through a data report function incorporated in the data station (Chromeleon or equivalent one). The reported data includes the compound name, retention time, and concentration.

9.0 Quality Control

- 9.1 A calibration is performed prior to the field measurements and when changing major components, including GC columns and the cold trap. The correlation coefficient (r) for each compound should be greater than 0.98. All calculations are automated through the data acquisition and processing software (Chromeleon or equivalent one).
- 9.2 Each analytical batch of 22 or fewer samples must include a one-point check standard and a blank.
- 9.3 The target compound concentrations measured during the analysis of the blanks must be below the compound's LOQ.
- 9.4 Calibration/one-point check standard
 - 9.4.1 A single point calibration (one-point check) is performed daily by analyzing the midpoint calibration standard (one-point check standard).
 - 9.4.2 The response of the present check standard must be within ± 20 percent of the previous check standard response. The daily run will be invalidated if the check standard criteria are not met, unless a valid explanation can be provided.
 - 9.4.3 A new calibration curve is required if the one-point check exceeds the

acceptance criteria.

- 9.4.4 Breakthrough and carryover can be determined through the response of light VOCs and the baseline level, respectively. If breakthrough occurs, the coldtrap needs to be changed. If carryover occurs, the baseline of chromatograms of subsequent samples will be carefully evaluated to determine whether the carryover affect the quantification of analytes of interest. The current sequence will be modified to include additional blanks to clean the system.
- 9.5 Time series of target compounds
The time series of targeted compounds will be reviewed to check the overall performance of instruments regarding capturing the variation in ambient VOC concentrations. The chromatogram for individual samples, wherein the concentrations of targeted compounds show a sudden increase or decrease, compared to the immediately previous sample, should be checked to ensure that the correct peak is integrated. The threshold to trigger this check is site specific and should be determined based on the measurements made at the site and address the variations due to the atmospheric processing and emissions.
- 9.6 Flow rate and sampling volume
 - 9.6.1 The sampling volume and duration for each sample is reviewed while conducting the time series review. The sampling volume and duration should be the same for each sample.
 - 9.6.2 Sampling flowrates should be checked monthly using a NIST traceable flowmeter. The flowrates between two checks should agree within \pm 10 percent. If the differences between two flowrate checks are greater than 10 percent, a new calibration will be carried out.

10.0 Maintenance

- 10.1 Change the upstream filter biweekly.
- 10.2 Check the water level of the hydrogen generator biweekly to ensure the water level is above the minimum line. Fill the water tank as necessary. The water is deionized water with resistivity $> 1 \text{ M}\Omega\text{m-cm}$, such as Milli-Q water.
- 10.3 Check the pressure of N_2 and He cylinder biweekly and change the cylinder when the pressure is below 150 psi or as necessary.

11.0 References

Northern Laboratory Branch Quality control manual, Revision 4.0, September, 2018
(<https://ww2.arb.ca.gov/sites/default/files/2018-10/nlbqcm.pdf>)

Northern Laboratory Branch Chemical Hygiene Plan

User guide for Thermo Scientific Trace 1300 and Trace 1310 Gas chromatographs.

Probe and monitoring path siting criteria for ambient quality monitoring. 40 CFR part 58, Appendix E.

Technical assistance document for sampling and analysis of ozone precursors, September 30, 1998. US Environmental Protection Agency

Appendix A: GC Flow System

Figures A1 and A2 illustrate the flow system of the GC. The blue arrows indicate the flow direction. The legends of C1-C3 refer to the chromatographic separation system with C1 as the TG1 column, C2 is as the column restrictor and C3 as the PLOT column. The legends of C4 and C5 refer to the auxiliary pressure control units of Aux4 and Aux5.

When a sample is injected for GC analysis, the sample is first separated by the C1 (TG-1 column), then enters into the flow splitter (a Dean switch). The earlier eluates are introduced to the PLOT column (C3) for further separation by turning on the Aux4. Then, after a period time (12.3 min in the current method), the Aux4 (C4) is turned off, and the Aux5 (C5) is turned on to divert the eluates from the TG-1 column (C1) to the restrictor column (C2).

There is always He flow through the C2 and C3 column regardless of the status of C4 and C5. This continuous He flow allows the elution of the analytes from both C2 and C3 columns for subsequent measurements by their respective FID.

Figure A1. Flow diagram of GC system with Auxiliary 4 on.

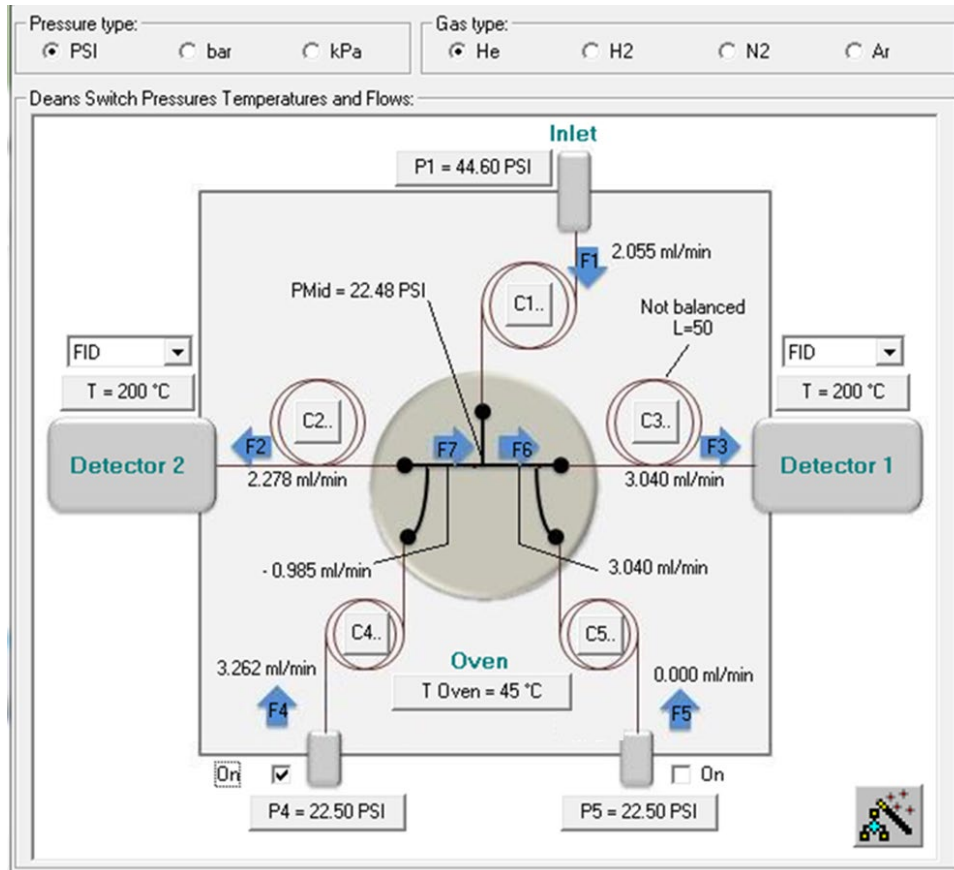
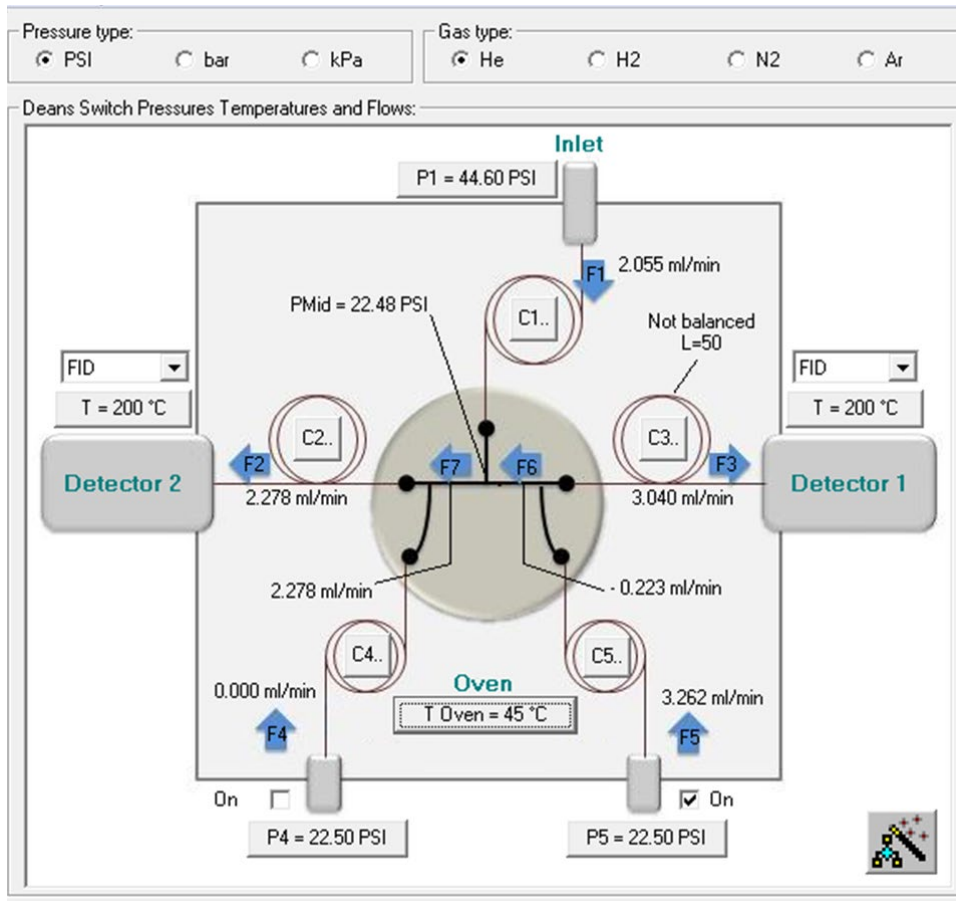


Figure A2 Flow diagram of the GC system with Auxiliary 5 on.



Appendix B: Flow Diagram of the Sampling System

Figure B1 shows the flow diagram during sampling when the CTD system is isolated from the analysis system (GC). The analysis and sampling systems are isolated and connected through the 3-way valve #1. The 3-way valve #2 is used to connect and isolate the Kori system from the adsorbent tube for VOCs.

Figure B2 display the flow diagram of the sampling during the thermal desorption and injection. The 3-way valve #1 connects the sampling system with the analysis system. Under this mode of operation, the 3-way valve #2 isolates the Kori trap from the sampling adsorbent tube, which allows the water collected by the Kori trap during the sampling to be purged out of the Kori trap and ready for the next sample.

Figure B1. Flow diagram of the sampling system on the sampling mode

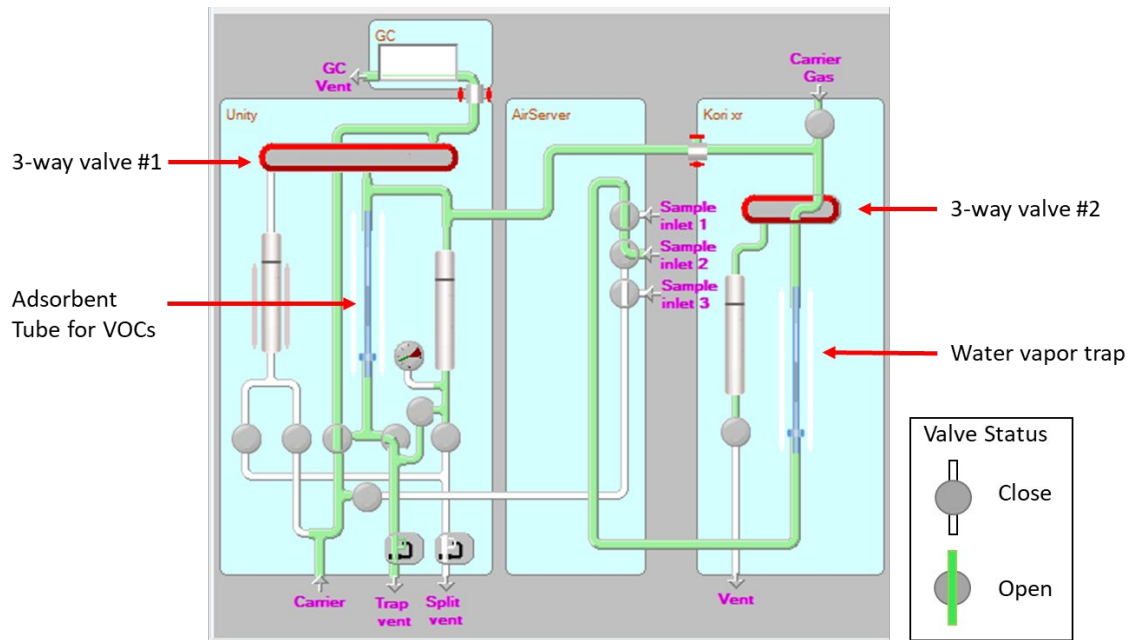
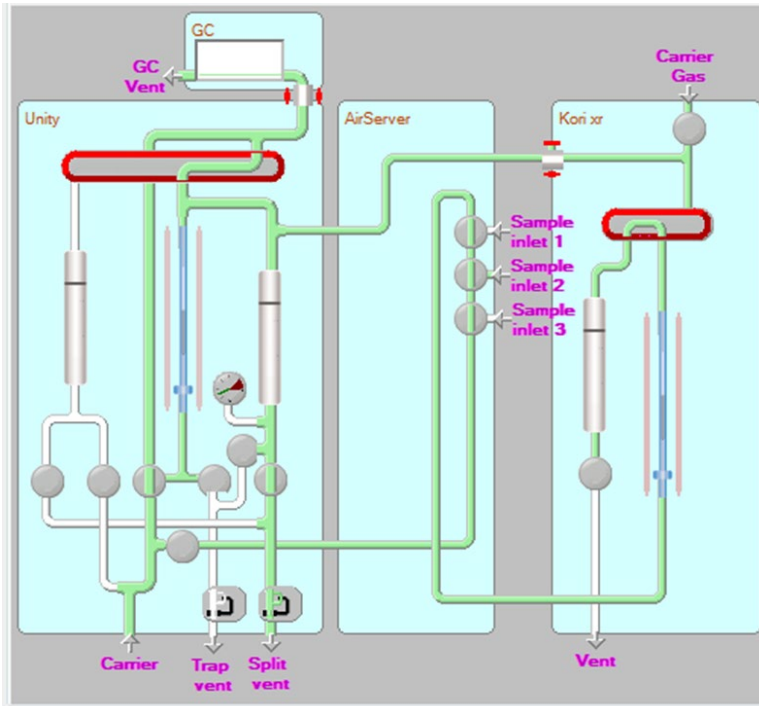


Figure B2. Flow diagram of the sampling system during the operation of thermal desorption and injection.



Appendix C: Instrument Software and Analytical Method

This appendix describes the typical method used by AMTS to measure atmospheric VOCs using a Thermo Trace 1300 GC-FID coupled with a Markes Air Server-Kori-Unity system.

The instrument software used for GC-FID is Thermo Chromeleon 7.2.7. The instrument software used for the sampling and desorption system is Markes Instrument Control 1.3.

GC-Method

Inlet

Injection type	Splitless
Temperature	200 C
Flow control	Constant Pressure at 44.6 psi

GC Oven

Retention time (min)	Rate (°C/min)	Target Temperature (°C)
0	Run	
13	0	45
40	5	170
50	10	200
52	Stop run	

Dean Switch

	time (min)	Status
Aux 4	0	on
	12.3	off
	time (min)	Status
Aux 5	0	off
	12.3	on

FID

Temperature	200	°C
Air Flow	350	ml/min
H₂ Flow	35	ml/min
N₂ Flow	40	ml/min

Markes Method

General

Coldtrap	U-T20PAM-2S	
Standby split on	2	ml/min
Flow path temperature	150	°C
GC cycle time	60	min
Run overlap	Yes	

Sample Purge

Sample purge time	1	min
Split flow	50	ml/min

Sampling

Sample by volume	yes	
Sample volume	600	ml
Sample flow	20	ml/min

Line Flush

Line flush time	1	min
Line flush flow	50	ml/min

Trap Setting

Trap purge time	1	min
Trap purge flow	50	ml/min
Trap low temperature	-30	°C
Trap heat rate	max	°C/s
Trap high temperature	280	°C
Trap desorb time	6	min
Desorb split flow	10	ml/min

Kori Setting

Trap low temperature	-30	°C
Trap heat rate	max	°C/s
Trap high temperature	300	°C

Appendix D: Targeted Compounds

CAS	Compound Name
71-43-2	Benzene
106-97-8	Butane (n-Butane)
98-82-8	Cumene (Isopropylbenzene)
110-82-7	Cyclohexane
124-18-5	Decane (n-Decane)
74-84-0	Ethane
100-41-4	Ethylbenzene
74-85-1	Ethylene (Ethene)
622-96-8	4-Ethyltoluene (p-Ethyltoluene)
142-82-5	Heptane (n-Heptane)
110-54-3	Hexane (n-Hexane)
75-28-5	Isobutane (2-Methylpropane)
78-78-4	Isopentane (2-Methylbutane)
108-67-8	1,3,5-Trimethylbenzene
95-63-6	1,2,4-Trimethylbenzene
540-84-1	2,2,4-Trimethylpentane
111-84-2	Nonane (n-Nonane)
111-65-9	Octane (n-Octane)
109-66-0	Pentane (n-Pentane)
74-98-6	Propane
115-07-1	Propylene (Propene)
100-42-5	Styrene
108-88-3	Toluene
108-38-3, 106-42-3	Xylenes (m & p)
95-47-6	Xylene (o)
74-86-2	Acetylene (Ethyne)
590-18-1	cis-2-Butene
624-64-6	trans-2-Butene
287-92-3	Cyclopentane
141-93-5	Diethyl benzene - M (m-Diethylbenzene)
105-05-5	Diethyl benzene - P (p-Diethylbenzene)
75-83-2	2,2-Dimethylbutane
79-29-8	2,3-Dimethylbutane
565-59-3	2,3-Dimethylpentane
108-08-7	2,4-Dimethylpentane
112-40-3	Dodecane (n-Dodecane)

CAS	Compound Name
620-14-4	3-Ethyltoluene (m-Ethyltoluene)
611-14-3	2-Ethyltoluene (o-Ethyltoluene)
592-41-6	1-Hexene
106-98-9	1-Butene
78-79-5	Isoprene (2-Methyl-1,3-butadiene)
526-73-8	1,2,3-Trimethylbenzene
108-87-2	Methylcyclohexane
96-37-7	Methylcyclopentane
592-27-8	2-Methylheptane
589-81-1	3-Methylheptane
591-76-4	2-Methylhexane
589-34-4	3-Methylhexane
565-75-3	2,3,4-Trimethylpentane
96-14-0	3-Methylpentane
107-83-5	2-Methylpentane (isohexane)
109-67-1	1-Pentene
627-20-3	cis-2-Pentene
646-04-8	trans-2-Pentene
103-65-1	Propyl benzene (n-Propylbenzene)
1120-21-4	Undecane (n-Undecane)