Standard Operating Procedure

for

Cleaning Regular and Silico-Canisters Using Xontech 960

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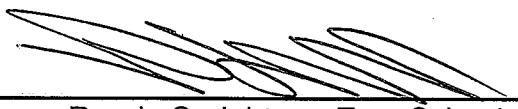
10/1/2009

Document Owners: Manfredo Giaccio and Jerry Knight, Env. Sci. 3 Date



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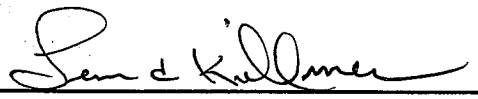
Process Owner: Randy Creighton, Env. Scientist Supervisor Date



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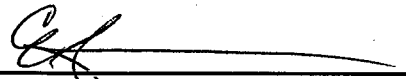
Approved By: Leonard Killmer, Env. Scientist Manager Date



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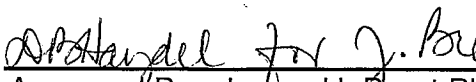
Approved By: Elaine Sorbet, Quality Assurance Officer Date



10-1-09

Approved By: James H. Brent PhD, Division Administrator Date

Annual Document Reviews:

Changes made, if any:

1st Review: Corrected errors; added the sub-section of batching on 08/15/2005.

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2nd Review : Made some minor modification on 08/22/2006.

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3rd Review : Added a Notepad file to help batching; added a summery of canister cleaning history table for better record keeping; added contents in 2.0, 7.0, 11.0, 17.0 and 21.9; made some other minor modifications on 08 /22/2007.

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4th Review : Reviewed on 09/15/2008. No change has been made.

5 th Review : Section 22.1, clarified documentation requirements.

Changes Reviewed and Approved by:

	Analyst:	Supervisor:	Manager	QAO	Date:
1st	_____	_____	_____	_____	_____
2nd	_____	_____	_____	_____	_____
3rd	_____	_____	_____	_____	_____
4th	_____	_____	_____	_____	_____

Table of Contents

1.0 Identification of Test Method

This standard operating procedure has been developed for cleaning regular and silico-canisters using Environmental System Inc's (previous name is Xontech) eight and twelve canister cleaning systems based on EPA 600-R-98/161: Technical Assistance--Documentaton for Sampling and Analysis of Ozone Precursors.

2.0 Applicable Matrices

VOC free zero air or nitrogen.

3.0 Detection and Quantitation Limits

The analysis on the control sample is done with LDEQ 1026 "GC/FID" for ozone precursor analysis. The method detection limit (MDL) for the method is determined according to Title 40CFR, Part 136. MDLs for the target ozone precursors are typically 1 ppbC with several compounds probably over 1 ppbC but under 2 ppbC. The quantitation limits are 5 ppbC.

4.0 Scope and Application

This procedure is applicable to 6 liter and 15 liter regular (SUMMA and Silonite) or silico–canisters. A 6 liter canister containing 1000 ppmC of VOCs has been satisfactorily cleaned using this procedure.

5.0 Summary of Method

The systems clean canisters using three cycles. Each cycle consists of evacuating and filling the canisters with clean air or nitrogen depending on type of canisters. The final evacuation, high vacuum finish (HVF), vacuums canisters down to 35 mtorr to prepare the canisters for sampling. The dirtiest canister in a batch is selected as a control sample. The canister is filled with clean air and submitted for FID analysis. If the analytical results meet the criteria for total non-methane organic compounds left, the batch is validated and the canisters are released.

6.0 Definition of Terms

6.1 Absolute pressure – pressure measured with reference to absolute zero pressure, usually expressed in units of kPA, or psia.

6.2 Total non-methane organic compounds (TNMOC) –- the sum of ppbC of the targeted and untargeted compounds quantified.

6.3 Acceptance criteria -- specified limits placed on the amounts of hydrocarbons left in the canister being cleaned. Target ozone precursors: </=2 ppbC; TNMOC: </=20 ppbC.

6.4 Laboratory information management system (LIMS) -- software that makes laboratory data management easy and concise. This lab uses LABWORKS ES developed by Perkin Elmer, Inc.

6.5 Milliohm (mohm) – 1/1000 of an ohm. An ohm is a SI unit of resistance to the flow of an electrical current through a substance (e.g: distilled water. The mohm value is directly proportional to the purity of the water.) Highly pure water will have a resistivity in the mega ohm, not the milli ohm, range.

7.0 Interferences and Pretreatments

Interferences come from water, in-house zero air or nitrogen. High quality VOC free water must be used and also replaced regularly. Purity of In-house nitrogen or zero air will be tested by GC/FID or GC/MS.

8.0 Safety

8.1 For Lab Services Division's general policy, see SOP#1769.

8.2 Safety glasses and lab coats are required in all laboratory areas.

8.3 Canisters with pressures above 20 psig should be partially vented in a hood before processing.

8.4 Gloves should be worn when removing canisters and other hot items after the end of a run.

8.5 Standard precautions must be taken to prevent electrical shock.

8.6 Standard safety procedures must be followed when handling compressed cylinders.

8.7 Warning! Don't place a canister containing liquid in any system manifold. At 120 °C, we risk rapid boiling and development of excessive pressure. If the canister valve is closed, the system's relief valve cannot function risking an explosion.

9.0 Equipment and Supplies

9.1 Barcode readers "Symbol LS4278".

9.2 Zebra TLP2844 printer.

9.3 ONE XONTECH MODEL 960 Canister Cleaning Command Unit for 8 or 12 canister systems. It features:

9.3.1 A manifold with four canister connections immersed in an oven with a temperature controller in degrees Celsius set at 120 °C.

9.3.2 A rough pump and a high vacuum pump system.

Command Unit

9.3.3 A dial gauge pressure is measured with reference to atmospheric pressure. Zero gauge pressure is equal to atmospheric (barometric) pressure. If the pressure is positive, the scale of 0 to 30 psig is used; if the pressure is negative, the scale of 0 to 30 inches in Hg is used.

9.3.4 A Hastings vacuum gauge with a range from 0 to 1000 mtorr. Millitorr (mtorr) are defined as 1/1000 of a torr. A torr is a pressure unit used chiefly in vacuum technology being the pressure required to support one mm Hg (mercury) at 0°C.

9.3.5 The “Time Remaining” window displays the minutes left to completion.

9.3.6 The “Cycle Number” window displays the number of ongoing cycles

9.3.7 The “Start – Continuous” toggle switch allows the selection of automatic run vs. continuous run.

9.3.8 The “AIR – H_20” programming push wheel is used to set the first number of wet cycles.

9.3.9 The” DRY AIR” push wheel is used to set the following number of dry cycles.

9.3.10 The “MANUAL FILL – EVACUATE” toggle switch operates the MANUAL FILL MODE or the MANUAL EVACUATION MODE.

9.3.11 The “HIGH VACUUM FINISH” switch commands the high vacuum pump activation in the “DRY AIR” mode. When the system is operating, this switch must be ON (Red).

9.3.12 The” “POWER” switch turns the unit ON.

9.3.13 The humidifier trap provides the required moisture for the wet cycles. It must be checked at the beginning of the run.

9.4 MODEL 960 Canister Cleaning Auxiliary Units - additional units under control of the command unit. Each Auxiliary Unit holds four canisters. The twelve-canister system uses two auxiliary units. The eight-canister system uses one auxiliary unit. They feature the following items:

9.4.1 A manifold with four (4) canister connections immersed in an oven with a thermo – regulator in degrees Celsius set at 120 °C.

9.4.2 A “POWER” switch used to turn the unit ON.

9.5 One RM Environmental Systems, Inc. Canister Cleaning Unit for 8 and 12-canister systems.

9.6 Temperature controller AEOmega CN 9000A °F.

9.7 Brass and stainless steel ferrules of appropriate size.

9.8 Plastic tubing to fit canister’s connections.

10.0 Reagents and Standards

10.1 Gases

10.1.1 Air UHP Ultra Zero Grade from the gas network

10.1.2 Nitrogen UHP Ultra Zero Grade from the gas network

10.2 Reagent Water 18 Mohm

Auxiliary Unit

11.0 Sample Collection, Preservation, Shipment, Storage, and Sample Rejection Policy

The cleaned canisters are stored in a designated room air conditioned at 25 °C. Whenever a canister is checked out, the canister inventory form (Table 3) must be filled out. The canisters must be leak tested by the field operators before sampling. The canister pressures before and after sampling must be recorded in the chain of custody. The canister pressure before sampling must be <-28 inches in Hg. If not, the canister is leaking. The canister will be fixed and re-cleaned.

12.0 Quality Control

For each batch, the dirtiest canister is selected as a control sample. The canister is filled with dry clean air and submitted for FID analysis. If all the targeted compounds are less than or equal to 2 ppbC and TNMOC is less than or equal to 20 ppbC, the batch is validated and the canisters can be

released. If not, the second dirtiest canister is analyzed again. If the second fails again, troubleshoot the system and re-clean the batch.

13.0 Calibration

This section is deliberately left blank for reason of inapplicability.

14.0 Procedure

14.1 Create a Batch for Canisters to Be Cleaned

14.1.1 Verify that all of canisters for a batch are properly labeled with LIMS #.

14.1.2 Create a Notepad file that contains all LIMS # of the canisters in the batch (entering LIMS # either using a barcode reader or manually inputting). The file is saved as **Reservoir** in the local computer.

14.1.3 Select the dirtiest canister as a control sample. To do this, input the Notepad file **Reservoir** into LIMS to find the dirtiest canister. Double click on CR_Canister **in Folder View** in LABWORKS. In the next screen, select **External File** and click on **Browse**. Select file **Reservoir** from your local computer and then click on **View Selection**, followed by clicking on **Enter Selection**. **Canister Cleaning Report** will appear on the screen. This report lists total NMOC for all the canisters in the batch. The canister with the largest number of total NMOC will be the control canister. Note: if there is more than one canister that has the same total NMOC or the canisters in the batch are process canisters, simply randomly select one canister as the control sample.

14.1.4 Verify that all the canisters in the batch have finished with the assigned tests and are assigned with test code $Clean. To do this, input the Notepad file **Reservoir** into LIMS again. Select **Modify/Delete Sample** from **Maintenance** menu in LABWORKS. In the next screen, select **External File** and click on **Browse**. Select file **Reservoir** from your local computer and then click on **View Selection**, followed by clicking on **Enter Selection,** icon for **Modify selected samples, Multisample spreadsheets** and **Analysis order.** A spreadsheet will show the information whether or not the assigned tests are completed and the test code $Clean is assigned. If not, consult with the supervisor.

14.1.5 Select QA **Batching** from **QA/QC** menu in LIMS.

14.1.6 Click on the icon for **Specify new batches by sample**.

14.1.7 Select **External File** and click on **Browse.**

14.1.8 Select file **Reservoir** from your local computer.

14.1.9 Click **Enter Selections**, followed by clicking on **Enter Selection.**

14.1.10 Click on **…** for **All analyses assigned to samples.**

14.1.11 Click on test code **$Clean.**

14.1.12 Click on icon **Create new batches,** followed by clicking on **OK**.

14.1.13 Ensure that **Batch size #** is not smaller than **Number Samples** and then click on **OK**.

14.1.14 Point to the dirtiest sample and left click on it. This sample will appear in the cell of **QA Sample**. This sample will be automatically assigned with additional test code $CLTNMOC.

14.1.15 Click on **OK.** Now you have created a batch for canister cleaning. Print a hard copy of the batch and check for accuracy. Record the batch information in the **Canister Cleaning Worksheet** shown in Table 1.

14.2 Operation

14.2.1 Fill the worksheet shown in Table 1 beginning with the analyst initials, the date. Circle the system used and the number of canisters processed. Select the gas used and cross the one not in use.

14.2.2 Verify that the pressure in the line is 40 to 50 psig and the lines are appropriately open.

14.2.3 Verify that the humidifier contains enough water, up to the **fill line**. System 1 allows removal of the humidifier and therefore water can be filled directly. Systems 2, 3 and 4 require a siphon to change the water as necessary for maintenance.

14.2.4 Verify that the HIGH VAC FINISH switch is ON (red).

14.2.5 If the batch contains only regular canisters, use air and a setting: 2 Air-H2O, 1 Dry Air cycles.

14.2.6 If the batch contains regular and silico-canisters, use the same gas and settings but do not heat the silico-canisters.

14.2.7 If the batch contains only silico-canisters, use nitrogen with settings: 0 Air-H2O cycles and 3 Dry Air cycles.

14.2.8 Connect all canisters to the pressure hoses. Do not over tighten the fittings.

14.2.9 Close the ovens to be heated, top with the insulating covers and wrap the insulating “socks” around the gaps.

14.2.10 Pressure leak test: with the power still off, close all canister valves. Pressurize the system by opening the valve of a pressurized canister with a gauge reading of at least 15 psig and

then close the canister's valve. Wait 2 minutes and note the gauge reading again. Record the reading in the worksheet (Table 1). Any pressure drop of more than 0.5 psig should require troubleshooting guidelines.

14.2.11 Vacuum leak test:

- Set the push-wheel of the AIR/H_2O cycle to 0.
- Set the push-wheel of the DRY AIR to 0.
- Ser the HIGH VAC FINISH switch ON (Red).
- Turn the power ON. Leave all the canister valves closed.
- Press the the "evacuate button" ON. This will start a 30 minutes evacuation procedure.
- Wait until 22 minutes before the end of the cycle and take the reading in the Hastings vacuum gauge. Record the reading in the worksheet (Table 1).
- A final pressure should be less than 35 mtorr and must be less than 50 mtorr.

14.2.12 Once the system has been found leak proof, open all canisters.

14.2.13 Turn the heaters ON.

14.2.14 Turn the start switch ON.

14.2.15 After 2 hrs 58 min (2 min before the end of the process) the system will attain final vacuum. Record the pressure from Hastings Manometer in the worksheet.

14.2.16 Close all the canisters.

14.2.17 Remove the canisters from the ovens.

14.2.18 Fill the control canister with dry zero air and submit it for FID analysis.

14.3 Troubleshooting Guidelines

14.3.1 Check that all line connections are tight. If any are loose, retighten, and then retest. In pressure testing, SNOOP™ can be used to detect a leak by applying it on individual fittings. Bubbles will show at the leaking site when the system is pressurized. SNOOP should not be used on canisters due to the risk of contaminating them during the vacuum cycle.

14.3.2 From all the auxiliary units, choose the most remote auxiliary unit from the Command Unit. Disconnect the unit from the manifold line and cap manifold line. THEN, test the system again.

TABLE 1 CANISTER CLEANING WORKSHEET

Air Organics, LSD, LOUISIANA DEQ

Analyst	
Run Time, Date	
System	1 , 2 , 3 or 4
Gas/Pressure	Air, Nitrogen/
Settings (WetCyc/DryCyc)	2 /1 or 0/3
Trap	
HVFnsh	
Batch Number	$Clean-
Number of the Canisters Batched	8 or 12
Leak Check/Starting–Ending Pressure	
Vaccum Leak Check: mTorr/min left	
FinalVac (mtorr)	
The first Canister for Certification (LIMS #/Canister #)	
TNMOC of the first Certified Canister	
Certified by FID/Analyst	
Data File:	
The second Canister for Certification (LIMS#/Canister#)	
TNMOC of the second Certified Canister	
Certified by FID/Analyst	
Data File:	
Date of Canisters-Releasing	

Comments:

Reviewed by: Date:

14.3.3 If no leaks are found, the problem is isolated to Auxiliary Unit 2.

14.3.4 If the leak persists, it could be either Auxiliary Unit 1 or the Command Unit. (NOTE: Leaks can also be found in the valves of canisters. If the leak cannot be found, disconnect all canisters one by one; plug the lines, and then retest.)

14.3.5 Proceed as above closing the connection between the Command Unit and Auxiliary Unit 1 by first disconnecting the two units and capping the main manifold line. Retest for leaking. (EXCEPTION: It has been noticed on several occasions that all lines passed leak tests, but pressure would not stabilize. This problem was due to the vacuum pumps' valves incorrectly sealing. By starting the vacuum cycle and letting the pump motor run several minutes, then ending the cycle, the system probably sealed itself without any further leaks.).

14.3.6 By following a leak will be quickly isolated. This will indicate which auxiliary unit, or possibly the command unit, is leaking. When the leaking unit is isolated, leak test the individual lines. Remove the individual line from the manifold, plug the connection, and retest the unit.

15.0 Evaluation of Data, Reporting Results and Calculations

15.1 The FID results for the control sample in a batch must meet the criteria specified in 12.0. Record the analytical results in the worksheet (Table 1).

15.2 If the control sample doesn't meet the criteria in 12.0, select the second dirtiest sample in the batch and submit it for FID analysis. Record the result in the worksheet. If the FID meets the criteria, modify the batch, that is, remove the first control sample from the batch and designate the second dirtiest canister as the control sample.

15.3 If the second dirtiest control doesn't pass the criteria, trouble instruments and re-clean the batch.

15.4 Enter Results into LIMS

15.4.1 From **Results** menu in LIMS, select **Results Entry** and click on **Batch Numbers**. Enter manually the batch number.

15.4.2 Click on **Find** and **Check All**.

15.4.3 Click on **View Selections.** Verify that the batch corresponds to the Worksheet and then click on **Enter Selection**.

15.4.4 A window **Results Entry** opens. Right click on the first sample cell in $CLEAN column and select **Enter or load results** from the coming menu.

15.4.5 A new dialog box shows the headings: **Component Result Entry** for the given sample at the given location.

- Start date is to be changed only in the first sample of the batch.
- Start Time is to be changed only in the first sample of the batch.
- End date is automatically set by the computer. However there is a small discrepancy that can be corrected directly. Also this correction holds for the first sample of the batch. The rest of the batch is automatically dated.

15.4.6 Manually enter the TNMOC and LIMS # for the control sample. The results can also be entered automatically using a barcode reader. To do so, create two barcodes for TNMOC and LIMS# by going to **Create Bar Code List** in **Utilities** menu. Save the entries by clicking on **Store Results**. Continue this process for each sample in the batch to completion.

15.4.7 On the **Results Entry** form, there is a second column **$CLTMNOC**. There is only one entry (TNMOC) in this cell. Manually or automatically enter the number.

15.4.8 Stamp the canister tags with the cleaning date and release canisters.

16.0 Method Performance

Refer to 18.0.

17.0 Pollution Prevention

Refer to LSD's Lab Waste Disposal SOP_1197.

18.0 Data Assessment and Acceptance Criteria

The final absolute pressure in the canisters must be less than 50 mTorr.

The FID results for the control sample in a batch must meet the criteria specified in 12.0.

19.0 Corrective Action for Out of Control Data

Should the concentration be at or above the limit, a second canister from the batch will be analyzed by FID. If the second canister passes the criteria, release the canisters in the batch except the first canister that was selected for certification. Re-clean the canister. If the second canister fails again, troubleshoot the instrument, re-clean the entire batch.

20.0 Contingencies for Handling Unacceptable Data

This section is deliberately left blank for reasons of inapplicability.

21.0 Waste Management

Refer to LSD's Lab Waste Disposal SOP_1197.

22.0 Data and Records Management

22.1 The canister cleaning worksheet is clipped together with the FID quantification report and a full report from Results Entry, containing each sample in the batch and all methods used during the certification marked "Done". This record will be kept in Laboratory Services Division for 10 years. The information is also maintained by, and available through, Laboratory Services Division, or the LIMS.

22.2 For each cleaning system, a database file (Table 2) that summarizes the cleaning history is maintained. This file contains the information of date of cleaning, batch number, total number of samples, LIMS# of the control sample, initial concentration of the control and analysis results. This database serves as reference for gaining access to **Canister Cleaning Worksheets**. In column **TNMOC of Control Canister,** zero is assigned if all the canisters in the batch are process/voided samples.

22.3 Canister inventory forms (Table 3) are placed in the room for the cleaned canisters. The form must be filled whenever a canister is checked out. The first line shows an example how the form is filled out. The filled forms will be kept in the secretary's office (A-05) to keep track of the canister flow.

23.0 Tables, Diagrams, Flowcharts, and Validation Data

Tables and diagrams are inserted in this SOP. Data are validated by the supervisor or the manager.

TABLE 2 SUMMERY OF CLEANING HISTORY FOR SYSTEM 1

Cleaning Date	Batch Number	Total Number of Canisters	LIMS # of Control Canister	TNMOC of Control Canister	FID Results
3/1/07	151080	12	AK03684	161	4.61
3/6/07	151342	12	AK03689	1237	2.52

TABLE 3 CANISTER INVENTORY

Canister #	Site	Date Out	Print Name	Initials
10010	LSU	08/16/07	R. Bailey	*REB*

24.0 References

24.1 Technical Assistance Document for Sampling and Analysis of Ozone Precursors. EPA/600-R-98/161. Research Triangle Park, NC: U.S. Environmental protection Agency. 1998

24.2 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air. Compendium Method TO-12. Method for the Determination of Non-Methane Organic Compounds (NMOC) in Ambient Air Using Cryogenic Preconcentration and Direct Flame Ionization Detection (PDFID). EPA-600/4-89/017. Research Triangle Park, NC: U.S. Environmental Protection Agency. 1998.

24.3 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air. Compendium Method TO-15. Determination of Volatile Organic Compounds (VOCs) In Air In Specially-Prepared Canisters and Analyzed By Gas Chromatography/Mass Spectroscopy (GC/MS). EPA-625/R-96/010b. Center for Environmental Research Information, U.S. Environmental Protection Agency. Cincinnati Ohio: June 1999.

24.4 Cleaning and Certification of Specially Prepared Canisters for Air Sampling. Standard Operating Procedure #312. USEPA REGION 9 LABORATORY. Richmond, CALIFORNIA. Rev. #0. Date: 07/08/99.