

## HYDROCARBONS-SAMPLER MAINTENANCE

### Section III

#### LABORATORY RESPONSIBILITIES

## Approval Sign-Off Sheet

I certify that I have read and approve of the contents of this revision of the "Hydrocarbon-Sampler Maintenance - QA Plan, Section III, Laboratory Responsibilities" with an effective date of October 27, 2010. **Sign, date and return to the Ambient Monitoring Section Chief.**

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## **2.18.3 Hydrocarbons – Sampler Maintenance QA Procedure, Laboratory Responsibilities**

**Note:** The following is a list of "significant changes" from Revision 1.0.

1) QA updated per QAP/SOP 2.39 "Standard Operating Procedure (SOP) for Preparing Quality Assurance Plans/SOPs".

### **2.18.3.1 SAMPLER MAINTENANCE**

#### **2.18.3.1.1 OBJECTIVES**

The sampler maintenance described in this section applies to the sampling equipment used to draw in an electrically timed volume of air (non methane organic compounds (NMOC) hydrocarbons) associated with ambient air sampling.

#### **2.18.3.1.2 Sampler Equipment**

The ambient air sampler is an electrically powered (115 volt AC) sampling system that consists of a sampling pump, two to four event timer mechanisms which can be programmed to sample air seven days a week within a twenty-four hour time period and elapsed time meters.

#### **2.18.3.1.3 Sampler Check for Field readiness**

**2.18.3.1.3.1** Prior to being placed in operation, HC lab personnel or PPB Chemist will check that the air sampling equipment is field-tested to ensure there are no leaks from the sample inlet tube to each of the outlets.

**2.18.3.1.3.2** The sampling system should be tested to ensure each sampling outlet has consistent sampling volume.

**2.18.3.1.3.3** The electrical system (keypad timers, elapsed time meters and solenoids) checked and is in operating condition.

**2.18.3.1.3.4** Airflow check valves checked and in operating condition.

#### **2.18.3.1.4 Operational Procedures**

**2.18.3.1.4.1** The power to the sampling system is established and on.

#### **2.18.3.1.4.2 Timer Glossary**

##### **2.18.3.1.4.2.1 Mode Selector**

- MAN - Automatic operations are bypassed when in the manual mode. Load(s) can be manually switched "ON" or "OFF".
- AUTO - Position for automatic operation. Events can be manually activated or deactivated; the timer will resume automatic operation beginning with the next set point. Timer should be in "AUTO" or "MAN" mode at all times.
- SET - To set clock, program, or modify program
- REVIEW - To check the program

#### **2.18.3.1.4.2.2 On Indicator Light**

Flashes when the selector switch is set in “REVIEW” or “SET”. Glows steadily when normally “open” contacts are “closed” during automatic or manual operation. Does not glow when normally “open” during automatic or manual operation.

#### **2.18.3.1.4.2.3 Programming Buttons**

- CLEAR - Clears display entry
- CLOCK - Enter the time of day when in the “SET” mode.
- ON/OFF - To enter programming steps when in the “SET” mode, as well as manually controlling the timer from the “AUTO” or “MAN” mode.
- AM/PM - Designates the time as AM or PM.

#### **2.18.3.1.4.2.4 Other**

- RESET - To erase the entire program.

#### **2.18.3.1.4.3 Timer Programming Steps**

*Warning: Do not press RESET button while programming or the entire program will be lost.*

##### **2.18.3.1.4.3.1 Set or modify the time of day:**

- 1) Turn selector switch to “SET”.
- 2) Press the numbered button corresponding to the day. For example, if the day is Monday, press the “2” button.
- 3) Enter the current time of day. For example, if the time is 8:00 AM, press 800 and press “AM”.
- 4) For our example, the display will show “2 ‘8:00”.
- 5) Press “CLOCK” button.
- 6) The display will now show: “- --:- -.”
- 7) To verify that the correct time is set turn the selector switch to “AUTO” or “MAN”. If the time is incorrect repeat steps 1-6.
- 8) Turn selector switch to “SET”

##### **2.18.3.1.4.3.2 Prepare “ON” and “OFF” set points for programming**

- 1) List all desired ON and OFF set points as shown in the example.

Example:     Load to be “ON” Monday at 8:00 AM  
              Load to be “ON” Thursday at 12:15 PM  
              Load to be “OFF” Monday at 8:05 AM

Prepare your list:

<u>DAY</u>	<u>TIME</u>	<u>AM/PM</u>	<u>ON/OFF</u>
2	8:00	AM	ON
5	12:15	PM	ON
2	8:05	AM	OFF

2) Now list all days to be repeated (exactly) from days already programmed.

Example: Tuesday to repeat Monday events.

Wednesday to repeat Monday events.

Prepare your list:

<u>DAY TO REPEAT</u>	<u>ON</u>	<u>DAY BEING REPEATED</u>
3	"ON"	2
4	"ON"	2

NOTES:

1. Any day can repeat any programmed day.
2. If a day already has a program, it cannot repeat another days cycle unless the scheduled events for that day are removed.
3. If a day being repeated has a program change, the days repeating it will recognize that change **Automatically**.

### 2.18.3.1.4.3.3 Enter the program

1) Enter the program in the exact order. Day; Time; AM/PM; ON/OFF

Example: for listing on/off set pts.

Press	5	12:15	PM	ON
Display	5--:--	5 12:15	5,12:15	--:--
Press	2	800	AM	ON
Display	2--:--	5 8:00	2, 8:00	--:--

NOTE: AM is indicated as " " flashing .



PM is indicated as " , " flashing.



2) Enter the program for repeat cycles.

Example: for listing on/off set pts.

Press	2	ON	1
Display	2 --:--	2 --:--	--:--
		flashing	↑
Press	3	ON	1

Display 3 --:-- 3 --:-- - --:--  
flashing →

- 3) The program is now complete. It is suggested that the program be reviewed before activating the loads. Follow the procedure shown in “REVIEW”.

#### 2.18.3.1.4.3.4 Review

- 1) Turn the selector switch to “REVIEW”
- 2) Press the button corresponding to the day being reviewed.
- 3) The display will show one of the following:
  - Example: Press 3 3 -2:-- (Tuesday “3” repeats Monday “2”)
 Or
  - Example: Press 2 --:-- (Monday events can be reviewed). In the second example, each time the “ON” button is pressed, the correct time for that operation will appear. Likewise each time “OFF” is pressed, off times will be displayed.

To modify a set point, that set point must first appear on the display. Next, the clear button is pressed. Turn the selector button to “SET” and enter the new information in the correct order (day, time, AM/PM, ON/OFF).

#### 2.18.3.1.4.3.5 Counting Set Points

Count each on and off set point. The example would total 3. Do not count repeat daily schedules. Up to 28 on or off set points can be programmed. The display would show “E EE:EE” if you tried to program the 29<sup>th</sup> set point.

#### 2.18.3.1.4.3.6 Operation

- 1) Turn selector switch to “AUTO”. **The sampler must be in the AUTO mode to collect samples automatically.**
- 2) Apply voltage to loads

NOTE: When in automatic operation, if the “ON” or “OFF” buttons are pressed, the loads will activate accordingly. The load will stay in this condition until the next programmed event.

#### 2.18.3.1.5 Sampler Installation

If sampler checks out satisfactorily, the PPB chemist or HC lab technician will contact the Electronics Calibration Branch to install the sampler at the site.

#### 2.18.3.1.6 Preventive Maintenance

##### 2.18.3.1.6.1 Sampler Leak Check

- 1) Check unit by pressurizing sampler with house air and using leak detection solution to locate leaks. Leak check housing intake unit, solenoid and connections. Each side of the sampler is checked separately.

- 2) Clean air is pumped through the sampling system for 24 hours to clean pump and sampling lines.

#### **2.18.3.1.6.1 Timers**

- 1) Program timer and collect a three-hour sample of clean zero air. Analyze canister sample to determine system cleanliness and timer accuracy.
- 2) Should at any time there is a timer that is not working properly, make a note on the sampling data form and contact ECB.
- 3) Timers have a battery back up to provide power incase there is a power outage. Replace batteries as needed.

#### **2.18.3.1.6.2 Elapsed Time Meters**

- 1) From **2.18.3.1.6.1** 1), check elapsed timer accuracy.
- 2) Should at any time there is an elapsed time meter that is not working properly, make a note on the sampling data form and contact ECB.

#### **2.18.3.1.6.3 Stainless Steel Tubing / Fittings**

- 1) Should at any time there is a fitting(s) that will not thread properly, make a note on the sampling data form and contact ECB.

#### **2.18.3.1.6.4 Check Valves**

- 1) 2 micron frit and airflow check valve replaced as samplers are returned after sampling period is completed (yearly).



FIGURE 1 NMOG Sampler

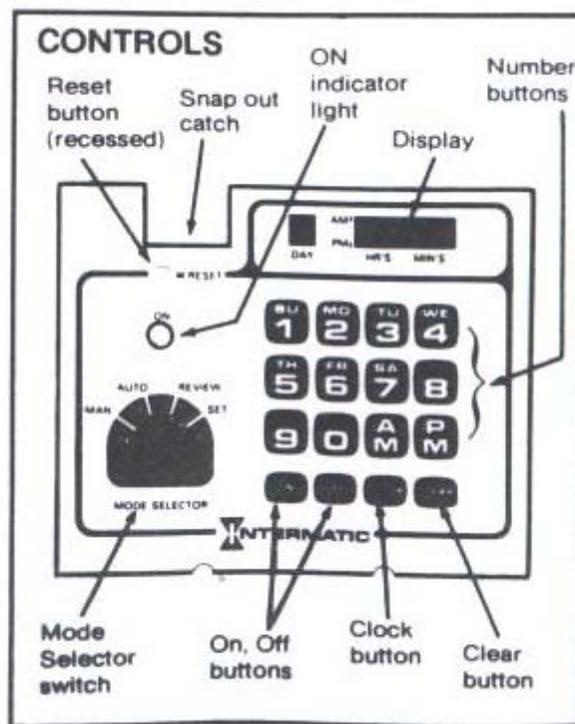


FIGURE 2 Electronic Seven Day Timer

**Table 1 HC Sampler Log**

<b>Sampler ID</b>		<b>Keypad SN</b>	<b>Elapsed Timer SN</b>	<b>Solenoid SN</b>	<b>Pump SN</b>	<b>Comments</b>
	Top L					
	Bottom L					
	Top R					
	Bottom R					
	Top L					
	Bottom L					
	Top R					
	Bottom R					
	Top L					
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## HYDROCARBONS-CANISTER MAINTENANCE

### Section III

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## **2.18.3 Hydrocarbons – Canister Maintenance QA Procedure, Laboratory Responsibilities**

**Note:** The following is a list of "significant changes" from Revision 1.0.

1) QA updated per QAP/SOP 2.39 "Standard Operating Procedure (SOP) for Preparing Quality Assurance Plans/SOPs".

### **2.18.3.2 CANISTER MAINTENANCE**

#### **2.18.3.2.1 OBJECTIVES**

The equipment and maintenance described in this section is recommended for reliable and reproducible analytical results of specific compounds selected as target analytes (non methane organic compounds (NMOC) hydrocarbons) associated with ambient air monitoring.

#### **2.18.3.2.2 Canister Equipment**

A Summa canister is an airtight, stainless steel container with an inner surface that has been electro polished and chemically deactivated. This process of chemical deactivation is the "Summa" process. This process combines an electro-polishing step with chemical deactivation to produce a surface that is chemically inert. A Summa surface has the appearance of a mirror, bright and shiny. The hydrocarbon laboratory uses 6-liter Summa canisters to collect ambient air samples over time (usually 3 hours).

#### **2.18.3.2.3 Operational Procedures**

**2.18.3.2.3.1** All canisters should be checked to ensure they have/maintain a vacuum of at least 26 in Hg prior to being shipped to the field.

**2.18.3.2.3.2** All canisters that come in with less than 5 psig should be checked for leaks after sample analysis.

**2.18.3.2.3.2** If canister will not hold a vacuum during the cleaning process, pressurize canister, record date and check daily with pressure gauge. If canister will not hold pressure after seven days, replace valve and re-check canister for leaks.

**2.18.3.2.4 Preventive Maintenance**

- 2.18.3.2.4.1** The air-sampling canister needs to be vacuum/pressure checked to ensure there are no leaks from the sample inlet valve and fittings. Pressurize suspect canisters and use SNOOP (bubble) solution to find leaks. Tighten as necessary, all fittings and valves that make up the sampling assembly.
- 2.18.3.2.4** Visually inspect the 20 ga. wall, type 304SS. internally passivated by SUMMA electropolish process; fitted with basering-stand and valve guard/handle; equipped with one Nupro SS4H high vacuum ultra-clean bellows valve and ¼-inch SWAGELOK connecting fittings.

HYDROCARBONS- ANALYTICAL SYSTEM MAINTENANCE  
Section III

LABORATORY RESPONSIBILITIES

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### **2.18.3 Hydrocarbons – Analytical System Maintenance QA Procedure, Laboratory Responsibilities**

**Note:** The following is a list of "significant changes" from Revision 1.0.

1) QA updated per QAP/SOP 2.39 "Standard Operating Procedure (SOP) for Preparing Quality Assurance Plans/SOPs".

#### **2.18.3.3 ANALYTICAL SYSTEM MAINTENANCE**

##### **2.18.3.3.1 OBJECTIVES**

The gas chromatograph equipment and maintenance described in this section is recommended for reliable and reproducible analytical results of specific compounds selected as target analytes (non methane organic compounds (NMOC) hydrocarbons) associated with ambient air monitoring. The sample mixture is separated into individual components by their interaction with the capillary column stationary phase, using temperature-programmed gas chromatography.

##### **2.18.3.3.2 Gas Chromatograph Equipment**

An analytical GC system is required, complete with a temperature-programmable oven, and either an integrator or PC-based data acquisition /analysis software. Also required are other accessories, including analytical columns and the gases required for GC-FID operation.

The GC system used is a Perkin Elmer Ozone Precursor System equipped with a direct flame ionization detector (FID), ATD 400 air sampler and interfaced with an IBM-compatible PC loaded with P E Nelson Turbochrom software.

In a flame ionization detector, the sample is injected into a hydrogen-rich flame where the organic vapors burn producing ionized molecular fragments. The resulting ion fragments are then collected and detected. The FID is nearly a universal detector. However, the detector response varies with the species of (functional group in) the organic compound in an oxygen atmosphere.

For specific details of the process and operation, the operator is instructed to consult the equipment manual.

**The Ozone Precursor System should only be installed by a Perkin-Elmer service Engineer.**

##### **2.18.3.3.3 Operational Procedures**

**2.18.3.3.3.1** Install the heated transfer line between the ATD 400 and AutoSystem GC.

- 2.18.3.3.3.2** Connect the components to the Air Sampler accessory.
- 2.18.3.3.3.3** All electrical connections for the instrument, auto sampler and integrator/software will have been completed as required by the equipment manual.
- 2.18.3.3.3.4** Install the columns in the AutoSystem GC oven.
- 2.18.3.3.3.5** Determine the midpoint pressure and correct restrictor length.
- 2.18.3.3.3.6** AutoSystem gases: helium, hydrogen and air should be connected and flows set.

Gas	Supply Pressure (PSI)	Use	Flow Rate (ml/min)
Helium	60	Carrier	5
Hydrogen	30	FID Fuel	~ 80
Air	>60	Nafion Dryer	280
		FID Fuel	800
		ATD Power	Negligible
Nitrogen	100	Peltier Purge	>100

Flame ionization detectors use hydrogen as fuel. If the hydrogen is turned on without a column attached to the injector and detector inside the oven, hydrogen could diffuse into the oven creating the possibility of an explosion.

#### **2.18.3.3.4 Shutdown Procedure**

If a service or procedure requires the gas chromatograph to be shut down, it is imperative that the following service shutdown instructions are followed.

- 2.18.3.3.4.1** Ensure that the gas chromatograph has finished analyzing samples and no longer requires gas
- 2.18.3.3.4.2** Cool the heated injectors, detectors and ovens.
- 2.18.3.3.4.3** Turn off all gases and slowly depressurize the system.
- 2.18.3.3.4.4** Switch off the power at the gas chromatograph on/off switch.

#### **2.18.3.3.5 Maintenance Procedure**

- 2.18.3.3.5.1 Replace gases-** Helium should be the best commercial grade available for normal chromatographic operation as it is used for the carrier gas. Nitrogen only needs to be dry as it is used for the Peltier Purge. Cylinders (tanks) of compressed gases should be handled with caution. Take care not to kink or stress the gas lines. Cylinders should be firmly clamped in the upright position. Gas cylinders should preferably be located on a flat, level base. All gas connections must be properly

tested at installation. Helium and nitrogen cylinders should be changed when pressure reads 200 psig.

**2.18.3.3.5.2 Nafion Dryer-** The Perma Pure MD™-Series gas dryers are shell and tube moisture exchangers that allow the transfer of water vapor between two countercurrent flowing gas streams. The devices consist of an inner tube of Nafion® polymer surrounded by an outer tube of various inert materials. The water vapor is selectively absorbed into the walls of the inner Nafion tubes and transferred to a purge gas stream. This transfer is driven by the difference in the partial pressures of the water vapor on opposing sides.

Typically the sample flows through the inner tubes and the purge gas flows in the opposite direction within the shell. The purge gas stream should be -40°C dew point air, nitrogen or other gas. The dry gas entering the dryer at the sample outlet performs two functions:

- 1) Provides a medium to carry away water vapor from the sample
- 2) Creates a temperature gradient along the length of the dryer

Drying is a continuous, self-regenerating process. No routine maintenance is required as the dryer is ruggedly constructed of corrosion-resistant materials and have no moving parts. The system is mounted in a small, environmentally sealed enclosure and requires only electricity to operate.

Performance of the dryer is dependent upon the purge air, as long as the zero air is moisture free the more efficient the dryer.

Replacement: preferably at the start of each season by factory trained personnel.

**2.18.3.3.5.3 Sorbent Trap** – replacement on an annual basis is recommended or when there is a possibility it is a source of system contamination. The sorbent material is held in place by a glass wool plug at the entrance/exit end, and a glass wool plug and spring at the other end. During the collection and analysis cycles, the system pressure cycles from a vacuum to ~ 48 psi. This can cause eventual failure of the trap if the packing becomes loose. Material displaced from the trap will contaminate the sample lines within the ATD. The onset of failure of the trap is manifested in the degradation of peak shape on the BP-1 column (excessive tailing of peak), while the PLOT column peaks remain relatively sharp. In addition, there may be the loss of recovery of some compounds, especially the higher molecular weight hydrocarbons (C<sub>6</sub>, benzene and up).

**2.18.3.3.5.4 Columns** – replaced when there is no peak resolution of two predetermined compounds using the following:

### Retention Factor (k)

The retention factor (k) is another measure of retention. It is the ratio of the amount of time a solute spends in the stationary and mobile phases (carrier gas). It is calculated using Equation 1. The retention factor was previously called the partition factor or capacity factor. Since all solutes spend the same amount of time in the mobile phase, the retention factor is a measure of retention by the stationary phase. It is a relative measurement and is linear. For example, a solute with a  $k = 6$  is twice as retained by the stationary phase (but not the column) as a solute with a  $k = 3$ . The retention factor does not provide absolute retention information; it provides relative retention information. An unretained compound has  $k = 0$ .

#### Equation 1. Retention factor (k)

$$k = \frac{t_R - t_M}{t_M} = \frac{t'_R}{t_M}$$

where:

- $t_R$  = retention time of first peak
- $t'_R$  = adjusted retention time
- $t_M$  = retention time of unretained compound

### Separation Factor (a)

The separation factor is a measure of the time or distance between the maxima of two peaks. It is calculated using Equation 2. If  $a = 1$ , then the peaks have the same retention and co-elute.

#### Equation 2. Separation Factor (a)

$$a = \frac{k_2}{k_1}$$

where:

- $k_2$  = retention factor of first peak
- $k_1$  = retention factor of second peak

**Resolution (Rs)**- The higher the resolution the less the overlap between two peaks. Separation is only the distance or time between two peak maxima (a). Resolution takes both **Separation factor (a)** and the **width** of the peaks into account. It is calculated using Equation 3. Baseline resolution usually occurs at resolution number of 1.50, however, there is no baseline between the peaks. Numbers greater than 1.50 indicate there is

baseline between the peaks. Numbers less than 1.50 indicate there is some peak co-elution.

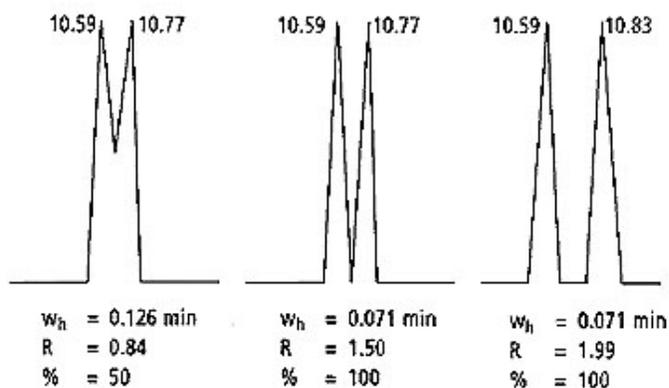
**Equation 3. Resolution ( $R_s$ )**

$$R = 1.18 \left( \frac{t_{R2} - t_{R1}}{W_{h1} + W_{h2}} \right)$$

Where

- $t_{R1}$  = retention time of first peak  
 $t_{R2}$  = retention time of second peak  
 $W_{h1}$  = peak width at half height (in units of time) of the first peak  
 $W_{h2}$  = peak width at half height (in units of time) of the second peak

**FIGURE 1 Resolution Examples**



**2.18.3.3.5.5 Column Bake out** – Periodically when ghost peaks or contaminants are detected on either the BP-1 or PLOT columns, method 3 is run for 1-2 hours to remove the contaminants.

**2.18.3.3.5.6 Data File Maintenance** - Periodically the Turbochrom data files should be transferred to a storage device (for example; zip disk or file server).

## HYDROCARBONS-CLEAN AIR SYSTEM MAINTENANCE

### Section III

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### **2.18.3 Hydrocarbons – Clean Air System Maintenance QA Procedure, Laboratory Responsibilities**

**Note:** The following is a list of "significant changes" from Revision 1.0.

1) QA updated per QAP/SOP 2.39 "Standard Operating Procedure (SOP) for Preparing Quality Assurance Plans/SOPs".

#### **2.18.3.4 CLEAN AIR SYSTEM MAINTENANCE**

##### **2.18.3.4.1 OBJECTIVES**

The clean air system and equipment described in this section are recommended for canister cleaning that produces air with <0.005-ppm hydrocarbons, carbon dioxide, methane, ozone, sulfur dioxide, hydrogen sulfide, ammonia, and oxides of nitrogen. The cleaning procedure involves purging the canisters with cleaned humidified air and then subjecting them to high vacuum.

The purpose of clean air system is to provide contaminant free air for canister cleaning. The canister interior surfaces should be free of contaminants and the canister should meet a predetermined cleanliness (i.e. < 10ppbC NMOC). This level of cleanliness minimizes the potential for carryover of organic pollutants from one sample to the next, and helps ensure that the samples collected are representative. Air from a standard oil-less air compressor will contain pollutants from the ambient air. In addition, various VOCs will be found in the compressed air because of the lubricants used in the air compressor. Hydrocarbon-free air may be purchased in cylinders and humidified before being used in the cleaning process. However, this approach may be cost-prohibitive.

##### **2.18.3.4.2 Clean Air System Equipment**

The AADCO pure air generator produces absolute clean air by passing an unclean source of air through a chromatographic like column where selective adsorption takes place and subsequent separation of the various components in the air. For further details of the process and operation, the operator is instructed to consult the equipment manual.

The AADCO pure air generator is calibrated for purity prior to shipment. The equipment is standardized for output purity and oxygen concentration against a factory standard to assure consistent performance between all pure air generators. This standardization is performed at an input pressure and output flow commensurate with the rated output flow of the generator being tested. To reproduce this output purity, the generator must not exceed the maximum permissible output flow for this unit and must have the proper input pressure and flow for same. Any output flow greater than the stated maximum output flow for any particular pure air generator produces air with purity worse than specifications.

### 2.18.3.4.3 Operational Procedures

- 2.18.3.4.3.1** Once all connections and checkouts have been completed as required by the equipment manual (Sections 3.0 thru 3.12 of the instrument manual), the pure air generator system is ready for connection to the equipment that is to receive the "zero " air. Connections must be made with 1/4 inch o.d. thin walled tubing with the using equipment located as close to the pure air generator as possible. 1/8 inch o.d. tubing or small-bore 1/4 inch tubing should be avoided because of the backpressure exerted on the system by the restrictive tubing.
- 2.18.3.4.3.2** If connection fittings at the using equipment are 1/8 inch swage type, use 1/4 inch large bore tubing from the pure air generator to the using equipment and then reduce to 1/8 inch tubing, keeping the 1/8 inch length as short as possible. All "tee's" should be 1/4 inch also.
- 2.18.3.4.3.3** After completing all plumbing connections close all needle valves and flow control devices at the using equipment and open the **Output Flow Adjust** valve, Figure 2, completely counterclockwise so that there is **NO** flow at the using equipment. The rotameter ball on the pure air generator should drop to "zero " indicating that the system is leak-tight. If not, check for leaks at all connections with some soap solution and tighten all loose connections until the rotameter ball does drop to "zero ". It is imperative that all leaks be detected and remedied before putting the system into full operation.
- 2.18.3.4.3.4** Output pressure from the pure air generator is set to provide enough flow to fill and maintain the pure air reservoir (65 psig) used in the canister cleaning process. The setting on the rotameter should not exceed 4.25 L/min as this flow setting equates to 10 L/min of actual flow, which is the capacity of the unit.
- 2.18.3.4.3.5** When the pure air generator is connected to external equipment (clean air reservoir), the **OUTPUT FLOW ADJUST** valve is opened counterclockwise, permitting flow through the valve. Flow control in this situation is performed at the external equipment through its flow control system. In this instance, the pure air generator is operating in a " back pressure" mode, placing the rotameter under pressure and no longer allowing the rotameter to be direct reading.
- 2.18.3.4.3.6** The pure air generator has a maximum output rating of ~4.25 L/min at 65 psig and the operator should know this before setting flows. **AVOID EXCEEDING THE OUTPUT CAPACITY OF THE PURE AIR GENERATOR AND JEOPARDIZE THE OUTPUT PURITY** (i.e., do not set the rotameter flow setting higher than 4.25 L/min).
- 2.18.3.4.3.7** Any instrument which is sensitive to even slight variations in oxygen concentration; i.e. flame ionization detectors used with total hydrocarbon analyzers, gas chromatographs, flame photometric detectors, etc., all

require incorporation of mixer receivers, **Figure 4**, in the pure air generator system for homogenization of the air mixture prior to use by the above-mentioned detectors. NDIR, chemiluminescent, photoionization, and electrochemical sensors are unaffected by slight variations in oxygen concentration and, therefore, do not require mixer-receivers.

#### 2.18.3.4.4 Shutdown Procedure

- 2.18.3.4.4.1** If the unit is not to be used for one week or more, depress the POWER switch, Figure 2, and immediately cap both the PURE AIR outlet (Figure 3) and the DUMP fitting (Figure 3) to avoid contaminants from entering the system.
- 2.18.3.4.4.2** Depress the PUMP switch. Unit is now ready for storage.

#### 2.18.3.4.5 Preventive Maintenance

- 2.18.3.4.5.1 Ballast Bleed** – All accumulated water is bled from the ballast tank at least once every three days during continuous operation. The installation of an automatic drain, Figure 2, located on the face of the pure air generator, does this operation. There should be a firm flow of air from the Ballast Bleed port, **Figure 2**, usually accompanied by water, which can be caught in a suitable container and discarded. The tank should be drained completely. During periods of high humidity the ballast tank should be bled every day.
- 2.18.3.4.5.2 Bleed System** – A weekly routine check should be made for plugging of the bleed system. In all cases, whether manual or electrical systems are used, when the bleed valve is opened there should be a strong flow of air from the Ballast Bleed port. If the air flow is weak or no water is emitted, there is a possibility that there is some blockage within the 1/4 " copper tubing leading to the plastic tubing leading to the bottom of the tank, the solenoid valve, or the 1/8 " plastic tubing leading to the front panel. This restriction should be cleared. Care should also be taken to be certain that the ballast bleed solenoid valve is operating, if that system is in the generator. Depressing the momentary contact switch should produce an audible " click " of the valve as well as a firm airflow from the BALLAST BLEED port.
- 2.18.3.4.5.3 Coalescing filter** – A visual check of the coalescing filter should dictate when the active ingredients should be changed. The change from orange to red indicates the effectiveness of the removal of moisture from the lab air is greatly reduced.

- 2.18.3.4.5.4 Methane Reactor** – A one or two minute observation of the methane heat lamp should reveal cycling of the lamp. This gives positive indication of heat control to the methane reactor. A glance at the pyrometer will also reveal that the unit is at temperature ( $290^{\circ}\text{C} \pm 10^{\circ}\text{C}$ ). If either check reveals some problem, see manual for remedial action.
- 2.18.3.4.5.5 Input Pressure** – A check of the INPUT PRESSURE gauge over a one or two minute period should reveal if the pressure switch is operating properly (75 psig). If not see manual for remedial action.
- 2.18.3.4.5.6 Purification Reactor** – A one or two minute observation of the rotameter should show the rotameter ball as remaining constant. If both the rotameter ball and the OUTPUT PRESSURE GAUGE show sudden drops during a one-minute cycle see manual (Section 13.8 for cause and remedy).
- 2.18.3.4.5.7** If the generator is equipped with a PURE AIR/ PURGE switch, switch to the PURGE position and monitor the purge flow on the rotameter. When the initial surge has diminished and the rotameter ball has reached a steady state condition, readings should be 10.0 minimum and 12.0 maximum for 5-LPM, 7.0 minimum and 9.0 maximum for 10-LPM. If not reading as per specs, see Section 13.21 of the instrument manual. Return the switch to the PURE AIR position upon completion of test.
- 2.18.3.4.5.8** If the generator is not equipped with a PURE AIR / PURGE switch, contact the factory for loan of a rotameter and instructions for its use to determine purge flows.
- 2.18.3.4.6 Routine Service Checks**  
Perform the following service checks routinely using the attached schedule, and procedures documented in **Table 1**. Checks may be performed more frequently but should be performed at least at the prescribed intervals. Also, **Table 2** (Monthly Quality Control Maintenance Check Sheet) should be completed weekly.
- 2.18.3.4.6.1 Daily Checks**
- 2.18.3.4.6.1.1 Output Pressure** - Output pressure gauge should indicate  $65\text{-psig} \pm 2\text{ psig}$  and should be constant throughout the cycling of the input pressure. Adjust, if necessary. Record the indicated output pressure weekly and every time an adjustment is made.
- 2.18.3.4.6.1.2 Output Flow**- Verify that the output flow meter is reading between 1-4 L/min, representing approximately 10 SLPM. Record the output flow weekly and every time an adjustment is made. Adjust, if necessary, the output flow using the procedure in Section 2.18.3.4.3.5.
- 2.18.3.4.6.1.3 Input pressure** – As the unloader valve cycles, the input pressure gauge will cycle between 55 and 75 psig. If the pressure is not cycling between 55 and 75

psig, adjust the valve using the equipment manual. Record the cycle pressures weekly and every time an adjustment is made.

**2.18.3.4.6.1.4 Methane Reactor** - verify that the indicator lamp is cycling on and off, indicating proper temperature control.

#### **2.18.3.4.6.2 Weekly Checks**

**2.18.3.4.6.2.1 Duty Cycle**- The 3-way solenoids at the input to the heatless air dryer columns are timed to energize alternately every 30 seconds. There is an audible click at the actuation. Time the duration each solenoid is energized and record the times in the spaces provided on the check sheet. The time for one complete cycle should be 60 seconds  $\pm$  2 seconds and there should be equal energize timing for each solenoid.

**2.18.3.4.6.2.2 Methane Reactor** - Record the temperature displayed by the methane reactor temperature measuring circuit. The temperature should be  $290^{\circ} \pm 10^{\circ}$ .

#### **2.18.3.4.6.3 Monthly Checks**

**2.18.3.4.6.3.1 Fans**- Verify the one fan in the reactor module is operating.

#### **2.18.3.4.6.4 Quarterly Checks**

**2.18.3.4.6.4.1 Solenoid Leak Check**- Disconnect the line from the auto water drain to the vent and cap both ends. Monitor the purge flow at the "DUMP" of the reactor module during the last 4 seconds of the 30-second cycle for each solenoid. There should be no flow during this period. Isolate the malfunctioning solenoid by disconnecting each electrical quick connect and repeating the test.

**2.18.3.4.6.4.2 System Leak Check (Methane Reactor Leak Check)** – Cap the "PURE AIR" outlet. Pressurize the system to normal pressures. At the end of the five minutes, observe the output flowmeter. The flowmeter should read zero flow. This procedure only checks the methane reactor and associated tubing downstream of the flowmeter. Several problems have been detected in this area.

**Table 1** Maintenance Schedule for the AADCO Model 737-12  
Pure Air Generator

	Daily*	Weekly	Monthly	Quarterly	Four Month Intervals
Input Pressure	X				
Output Pressure	X				
Output Flow	X				
Methane Reactor	X				
Duty Cycle		X			
Input Pressure Timing Cycle		X			
Drain Ballast Tank		X			
Q C Checklist		X	X		
Fans			X		
Discharge Unloader valve			X		
Solenoid Leak Check				X	
System Leak Check				X	

\* or each day the operator services the analyzer

Monthly Quality Control Maintenance Checksheet  
 AADCO Model 737-12 Pure Air Generator

Location: \_\_\_\_\_  
 Station Number: \_\_\_\_\_  
 Property Number: \_\_\_\_\_

Month/Year: \_\_\_\_\_  
 Technician: \_\_\_\_\_  
 Agency: \_\_\_\_\_

DATE	INPUT PRESSURE				OUTPUT PRESSURE		OUTPUT FLOW		TIMING		METHANE REACTOR TEMP
	LOWER		UPPER		AS FOUND	FINAL	AS FOUND	FINAL	DUTY CYCLE (SEC)	INPUT PRESS (SEC)	
	AS FOUND	FINAL	AS FOUND	FINAL							
									/	/	
									/	/	
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Operator Instructions:

- 1) Daily checks: Input pressure, Output pressure, Output flow, Methane reactor cycling, (record weekly) check chart traces
- 2) Weekly checks: Duty cycle timing, Input pressure cycle timing.
- 3) Monthly checks: Fans date checked: \_\_\_\_\_
- 4) Quarterly checks: Solenoid system leak check, date last checked: \_\_\_\_\_
- 5) As required: Replace auto water drain filter date checked: \_\_\_\_\_

DATE	COMMENTS OR MAINTENANCE PERFORMED

REVIEWED BY: \_\_\_\_\_ DATE: \_\_\_\_\_

**FIGURE 1 Monthly Quality Control Maintenance Checksheet**

**2.18.3.4.6.5 Flow Malfunctions**

<u>Problem</u>	<u>Probable Cause</u>	<u>Fix</u>
Low input pressure or inability to maintain proper input pressure	Problem with compressed air supply	Verify compressed air supply
Unable to vary output flow	Sticking flowmeter	Clean flowmeter
High output flow or flowmeter pegged	Leak in methanator	Check lines between methanator and flowmeter for leaks. If no leaks, replace methanator
Pump relief valve relieving	Auto-water drain filter plugged	Replace filter element in auto water drain

**2.18.3.4.6.6 Troubleshooting**

**2.18.3.4.6.6.1 General Information-** The manufacturer's Instruction Manual contains information pertaining to troubleshooting and should be your first source of information. Additional problems, which have occurred, are outlined below. Space is provided on the Monthly Q.C. Checksheet for recording malfunctions, causes, fixes, and actions taken to prevent reoccurrences.

**2.18.3.4.6.6.2 Electrical Malfunctions**

<u>Problem</u>	<u>Probable Cause</u>	<u>Fix</u>
Power fuse blown	Short circuit in methanator	Replace methanator
Momentary loss in output pressure at the end of each solenoid duty cycle	Solenoid timing problem	Check cam timing wiring

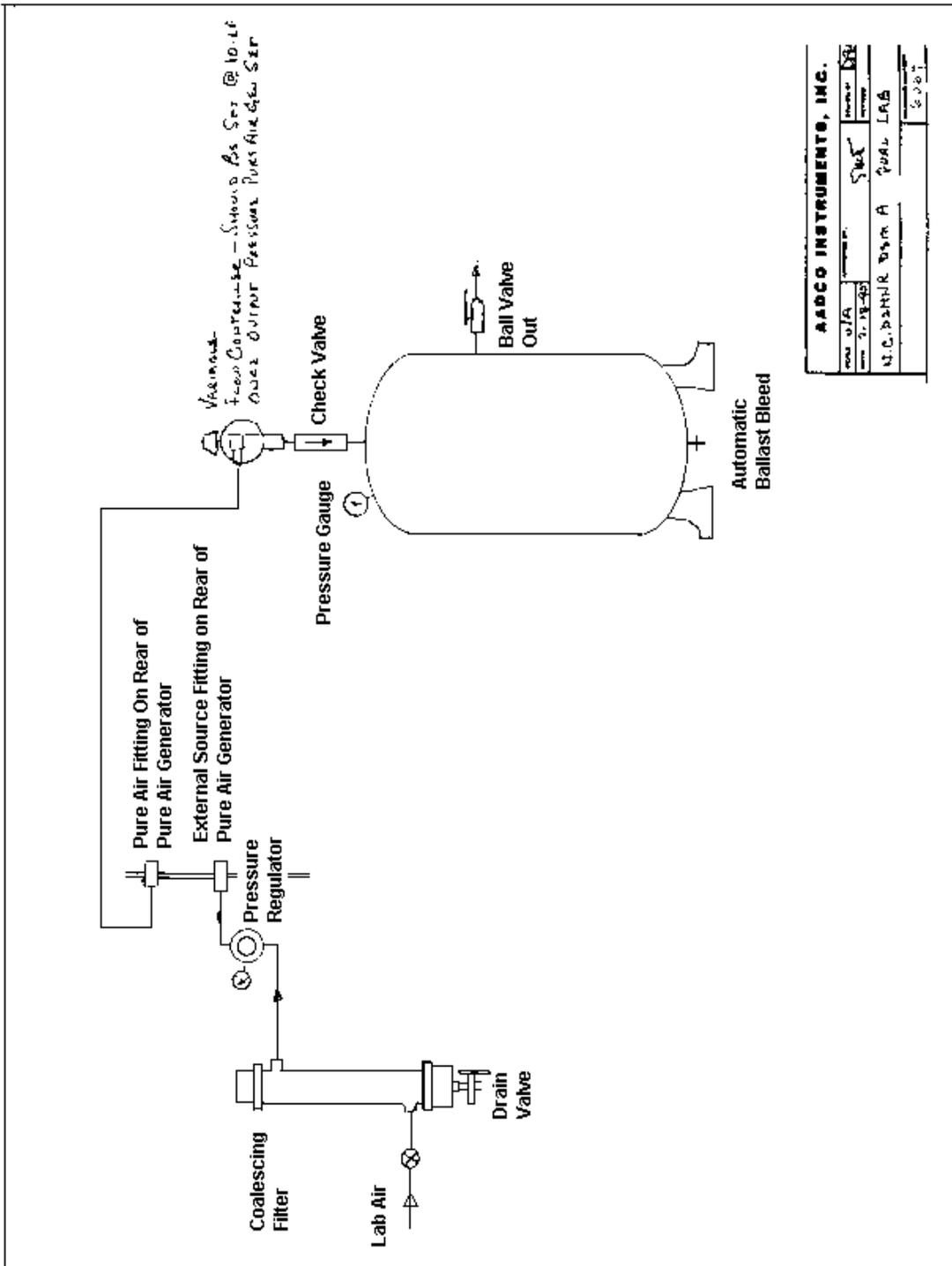
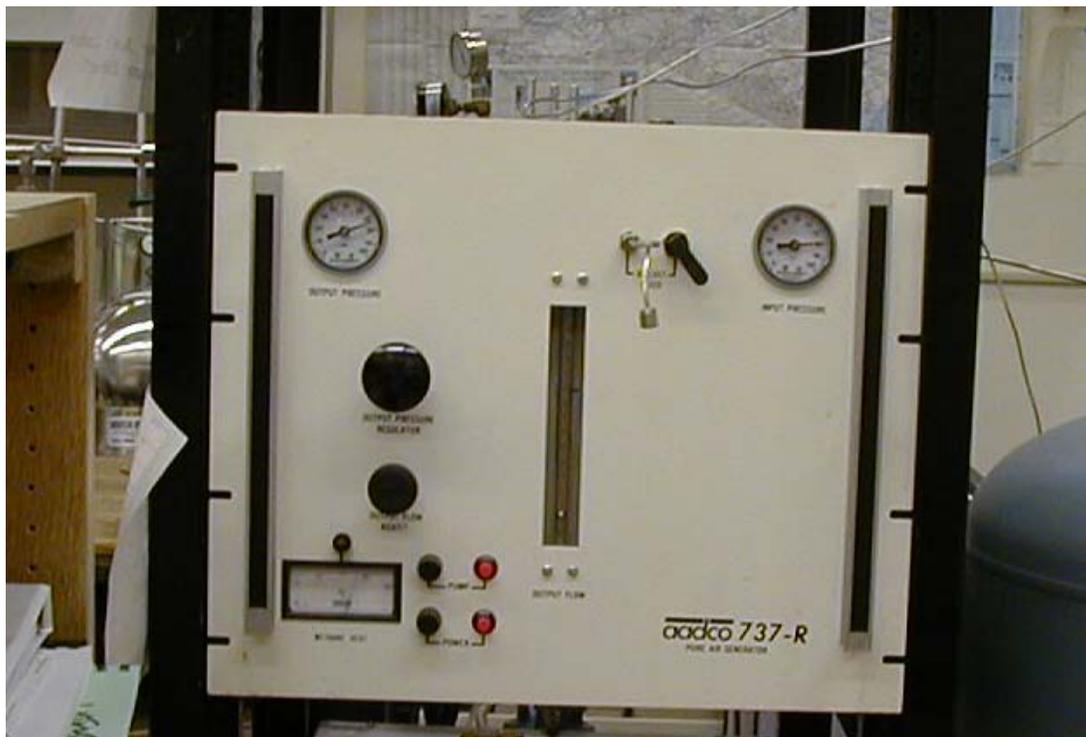
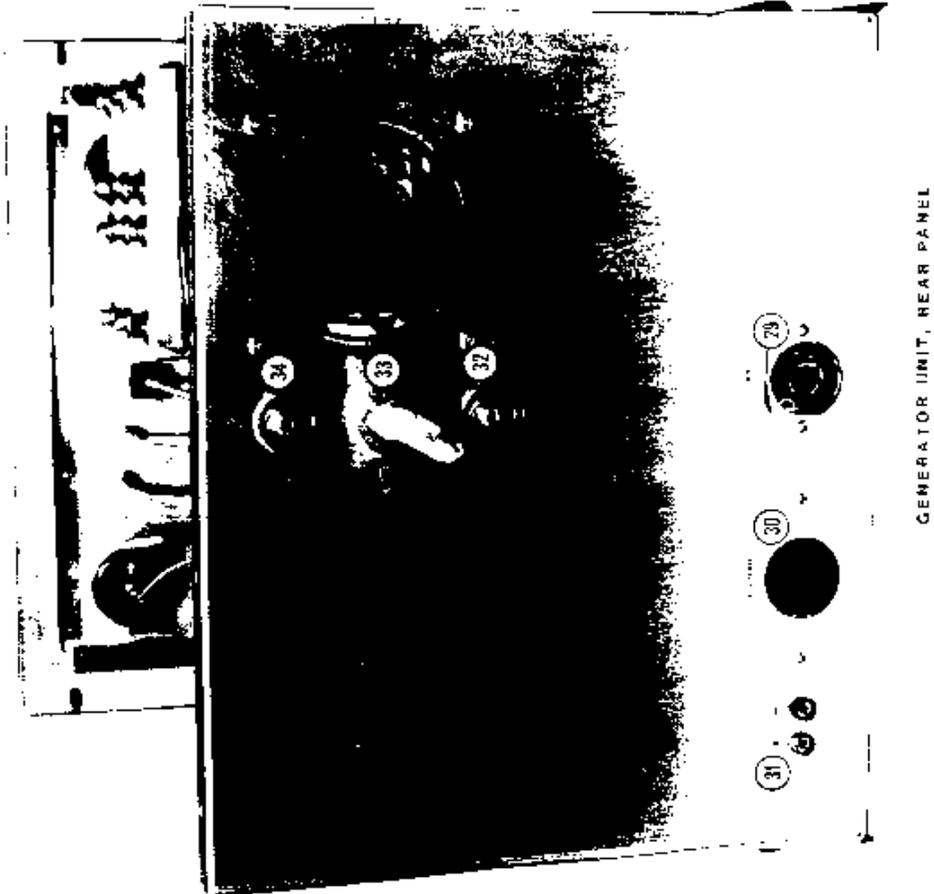


FIGURE 2 Generator Diagram

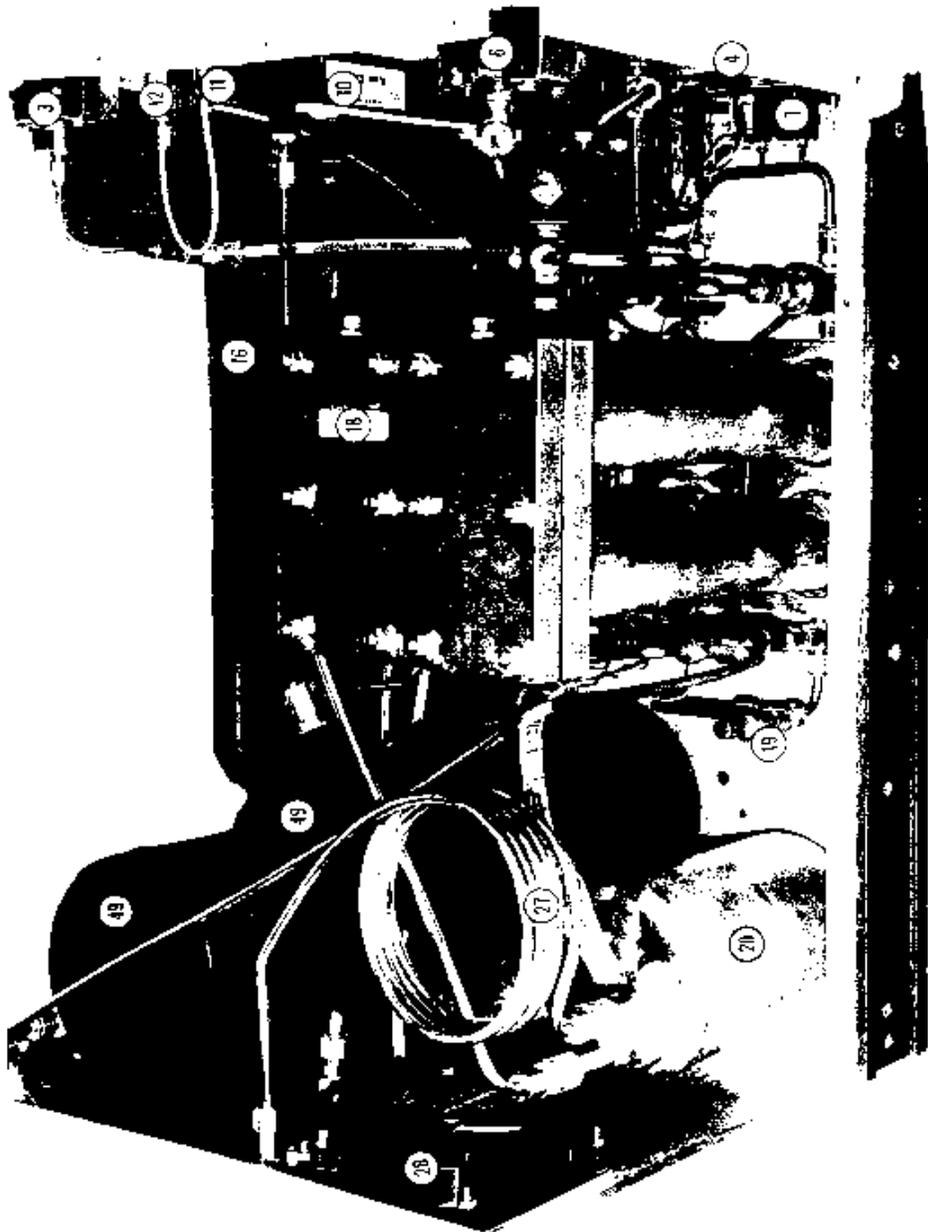


**FIGURE 3 Front Control Panel**



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FIGURE 4 Rear Control Panel



GENERATOR UNIT WITH MIXER-RECEIVERS

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FIGURE 5 Generator Unit With Mixer-Receiver

## HYDROCARBONS-HYDROGEN GENERATOR MAINTENANCE

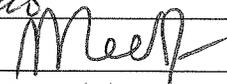
### Section III

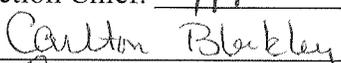
#### LABORATORY RESPONSIBILITIES

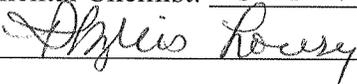
## Approval Sign-Off Sheet

I certify that I have read and approve of the contents of this revision of the "Hydrogen Generator Maintenance - QA Plan, Section III, Laboratory Responsibilities" with an effective date of October 27, 2010. **Sign, date and return to the Ambient Monitoring Section Chief.**

Joette Steger, PPB Supervisor: 

Donnie Redmond, Ambient Monitoring Section Chief:  6/30/11

Carlton Blakley, Environmental Chemist:  10/27/2010

Chemistry Technician:  11-3-2010

Chemistry Technician: \_\_\_\_\_

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### **2.18.3 Hydrocarbons – Hydrogen Generator Maintenance QA Procedure, Laboratory Responsibilities**

**Note:** The following is a list of "significant changes" from Revision 1.0.

1) QA updated per QAP/SOP 2.39 "Standard Operating Procedure (SOP) for Preparing Quality Assurance Plans/SOPs".

#### **2.18.3.5 HYDROGEN GENERATOR MAINTENANCE**

##### **2.18.3.5.1 OBJECTIVES**

The hydrogen generator system described in this section is a self-contained unit that can provide a constant flow of hydrogen for a gas chromatograph flame ionization detector.

##### **2.18.3.5.2 Hydrogen Generator Equipment**

The Nitrox hydrogen generator uses a special ion exchange membrane to produce a flow of ultra-pure hydrogen. Use of the electrolytic dissociation process enables water to be broken down into hydrogen and water. The oxygen is released into the air, while the hydrogen is retained to form the product flow. A long-life desiccant cartridge purifies the hydrogen even further so that it attains the desired grade of purity and ensures constant reproducible results.

For further details of the process and operation, the operator is instructed to consult the equipment manual.

##### **2.18.3.5.3 Operational Procedures**

- a.** Ensure that the power switch is in the off position. Connect one end of the power cord to the rear of the unit and the other end to a 20-amp 115v AC power source.
- b.** Switch generator on. The generator will give an audible indication of the start-up process, and all LEDs will illuminate for approximately two seconds.
- c.** During start-up, the unit may automatically revert to the last error mode it experienced. Press the black reset button to clear it and continue with the start-up procedure. If it cannot be cleared by this method follow the faultfinding procedure section in the instrument manual.
- d.** The start-up builds internal cell pressure up to 6.2 bar g. (indicated on the main display panel) using maximum current. This takes approximately ten minutes during which the system check LED will flash.

- e. The generator will continue the system check and a leak test takes place or two seconds. The system check LED will continue to blink.
- f. Outlet solenoid valve opens. The system check is now complete and if successful the LED becomes constantly lit.
- g. Set the outlet pressure regulator to 40 psig.

**Note:** The generator will now run maintaining flow at the set pressure. It is advised that the outlet tubing be purged for 15 minutes to remove oxygen before connecting to the application.

#### 2.18.3.5.4 Shutdown Procedure

If a service or faultfinding procedure requires the generator to be shut down, it is imperative that the following service shutdown instructions are followed carefully.

- a. Ensure that the gas chromatograph has finished analyzing samples and no longer requires gas.
- b. Switch the generator off at the on/off switch and isolate from mains supply by removing the appliance connector from the application inlet.
- c. Slowly depressurize the system. Watch the outlet pressure gauge on the front of the generator. Once the gauge shows zero, the valve can be fully opened. The generator is now shut down.

#### 2.18.3.5.5 Maintenance

- a. **Water Indicators** – To ensure continuous operation, regularly check the water level and top off if necessary.

There are four water level indicators on the front of the generator:

- Top green LED illuminated constantly: Water level full
- Central green LED illuminated constantly: Water acceptable level
- Central LED flashing: Water low, refill as soon as possible
- Red LED illuminated: Water empty, alarm condition, refill to restart

Before filling the water containers, always check the quality of the deionised water and ensure it falls between the tolerances outlined in the technical specification of the instrument manual.

**b. Water Conductivity Indicators** – The hydrogen generator has a built in conductivity meter with alarm to ensure the correct quality of deionised water is used at all times. There are ten LEDs located on the right hand side of the front display panel indicating the following:

- Seven green LEDs indicating good quality water
- Two amber LEDs indicating that the quality of water is near the limit for operation
- One red LED indicating that the quality of water could cause damage to the process. At this point the generator will shut down on alarm

The conductivity meter is there for two reasons:

- To ensure good quality water is used at all times.
- To give an indication of when the deioniser bags should be changed.

**c. Deioniser Bags** – There are two deioniser bags for each generator (one for each water container). The deioniser resin scavenges any remaining ions from the water and helps to stop the conductivity from rising during operation.

The bags should be replaced:

- If the conductivity is rising to alarm level when the same quality water is being used consistently.
- Every six months.

**d. Desiccant Cartridge Indicator** – The internally mounted desiccant cartridge is in the system to dry the hydrogen gas before feeding the application. The life of the cartridge will depend on the gas usage.

When the cartridge is new, the desiccant will be a mixture of orange and white material. Regularly check the window on the right hand side of the front door to see any color change. The flow of gas runs from top to bottom, therefore the color change (which turns the material to clear) will start from the top. Once the change is visible, order a refill kit (see instrument manual).

The desiccant does not need changing until the color change reaches the bottom of the window.

There are two ways of replacing the desiccant:

- Order the whole cartridge assembly.
- Order the refill kit and follow instructions (see instrument manual)

#### **2.18.3.5.6 Maintenance Procedure**

##### **a. Replacing Water**

If it becomes necessary to replace the water in the hydrogen generator, first switch off the unit by using the shutdown procedure. Find a watertight container that has a minimum volume of 4 liters and place it at a level that is below the base of the generator. Take the tube and drain connector supplied with the generator, open the door on the front of the generator and push the connector

into the drain bulkhead. Drain all of the water into the container and dispose of the contents. Never refill the generator with contaminated water that has been drained from the generator.

Remove the drain and tube from the bulkhead, take approximately 3-4 liters of fresh deionised water and refill both water containers to the full mark on the level indicator.

Follow restart procedure as described in the Section 2.18.3.5.7.

**a. Filling Water Containers**

If the illuminated water indicators are showing either the static green or flashing green LEDs illuminated, the water can be filled simply by leaving the generator running and removing the top caps from the containers. Top up the containers with fresh deionised water to the desired level ensuring not to exceed the maximum level shown in the water level window. Replace the container caps securely.

**b. Maintaining Non-Interrupted Operation**

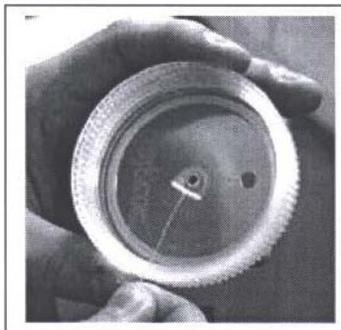
To ensure that the hydrogen generator runs continuously and to prevent alarm conditions occurring, regularly check the following:

Water quality  
Water level  
Desiccant cartridge  
Flow indication LEDs

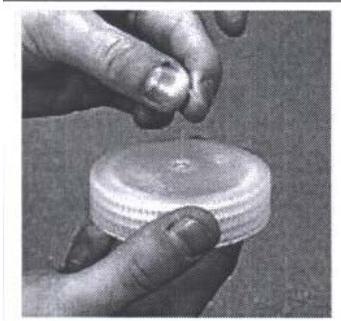
**c. Deioniser Bag Change**

*Installing Deioniser Bags*

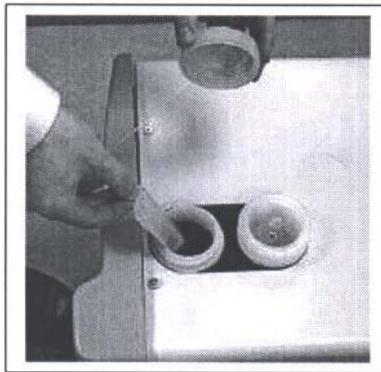
Remove both water tank caps. Take the two-deioniser bags out of their packing and push the locating "T" piece through the hole in the center of each cap (one deioniser bag in each cap).



Pull locating "T" fully through and hang bag onto the cap as shown.



Place one deioniser bag into each water container and replace caps securely



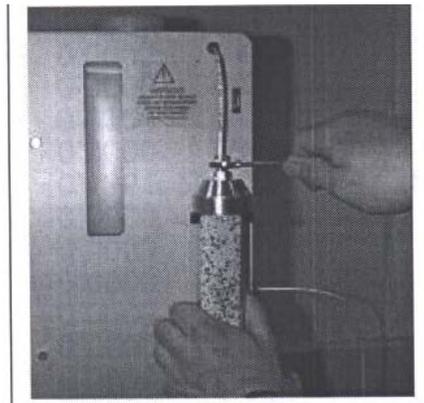
**d. Desiccant Cartridge Change**

**Note:** It is imperative that the service shutdown instructions are followed carefully PRIOR to commencement of any maintenance procedures.

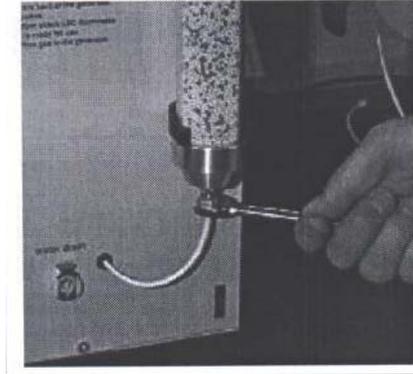
**Note:** Ensure that the generator has been fully depressurized before proceeding.

***Replacing Desiccant/Dryer Cartridge***

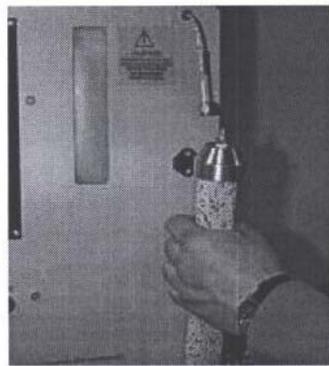
- 1) Open the door on the front of the generator. Loosen the tubing nut from the top of the desiccant cartridge by turning counter-clockwise and pull the tubing free.



- 2) Repeat the process with the tube fitting at the bottom of the cartridge.



- 3) Pull cartridge free from the mounting clips.

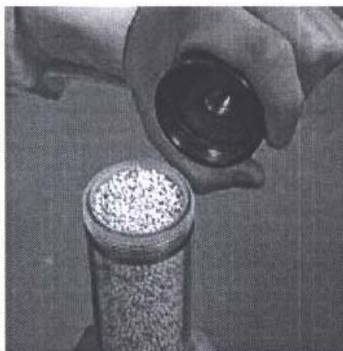


**Note:** If you are changing the whole cartridge assembly, the go to point 10.

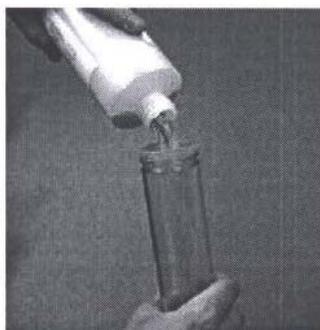
- 4) While holding the cartridge firmly and upright, remove one end by turning the end cap counter-clockwise.



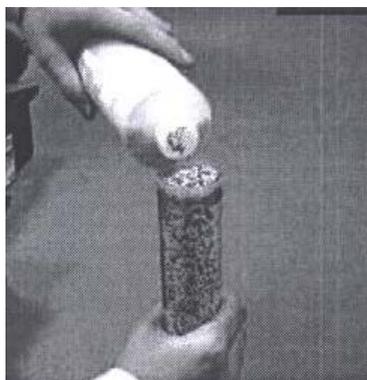
- 5) Completely remove end cap and keep in a safe place. Discard all of the used desiccant material. Do not regenerate the desiccant material. The cartridge housing is plastic and not suitable for regeneration.



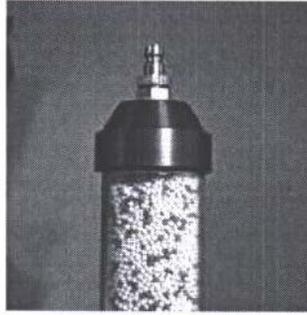
- 6) Using a clean cloth, carefully wipe the inside of the cartridge to remove any dust. Remove the cap from the desiccant refill kit and slowly pour the material into the cartridge.



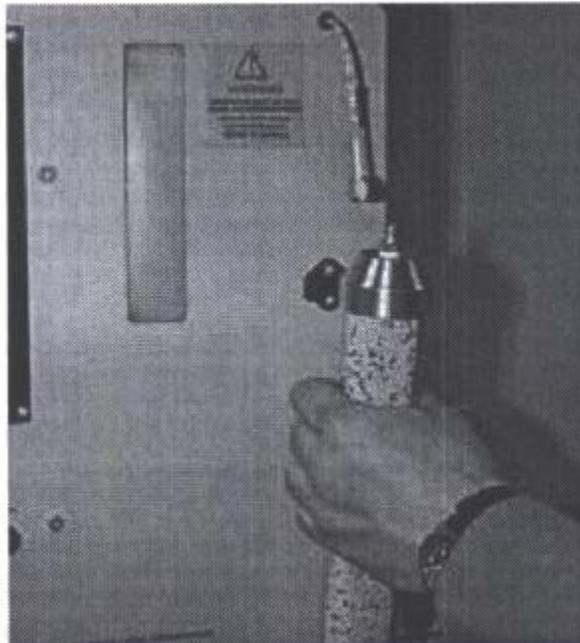
- 7) Fill to the top but be careful not to over fill.



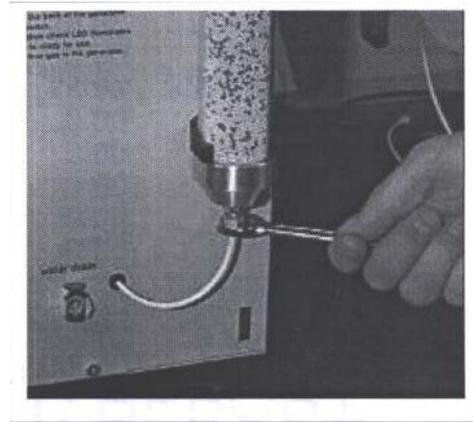
- 8) Replace cap firmly by turning clockwise. Ensure that the desiccant does not get trapped under the "O" ring seal on the end cap. If desiccant does get trapped, unscrew cap and remove some desiccant. Re seal.



- 9) Push the cartridge forward to locate into mounting clips.



- 10) Re-connect the flexible tubing to the top and bottom fittings on the desiccant cartridge.

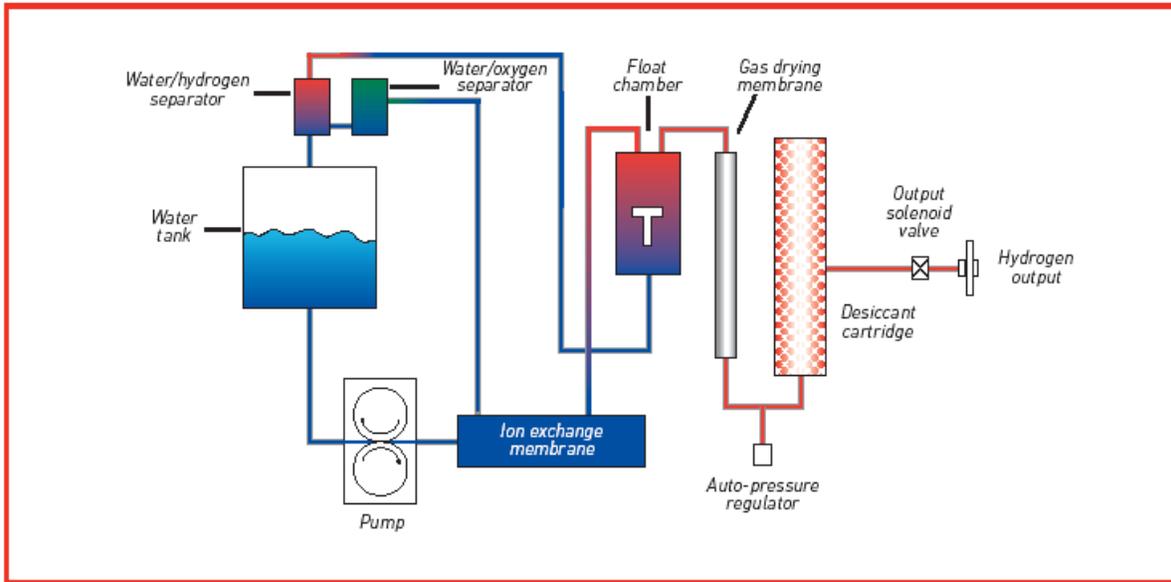


11) The desiccant cartridge is now connected

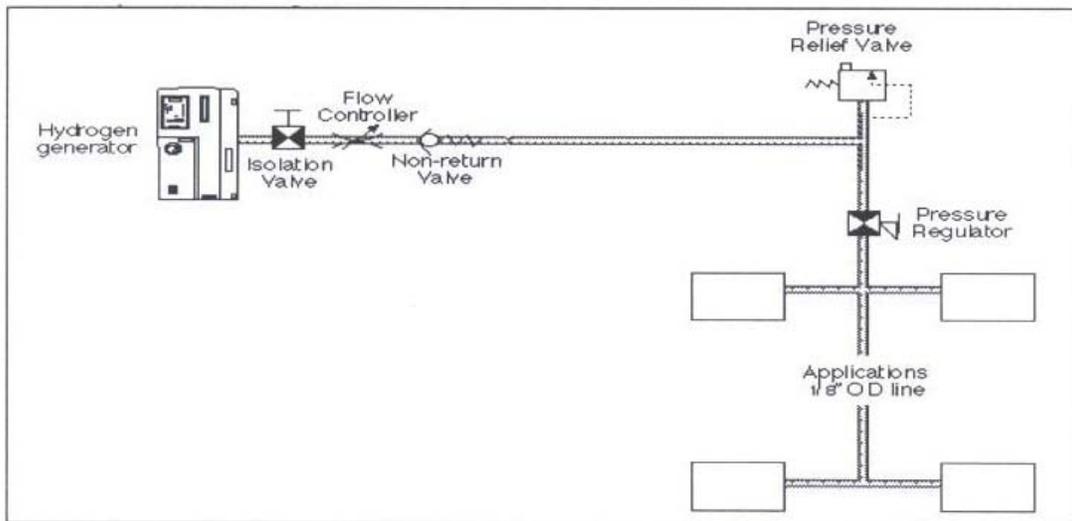
#### 2.18.3.5.7 Restart Procedure

- a. Ensure that any spare parts or covers have been replaced and fixed securely into position.
- b. Reconnect pipe work from the outlet of the recommended isolation shut off valve to the application and turn valve to the closed position.
- c. Reconnect main supply to the back of the generator.
- d. Switch ON using the on/off switch.
- e. Once the green flashing *system check LED* illuminates continuously, the generator is ready to use.
- f. Purge the system for 10 to 15 minutes to ensure purity and begin to supply application with gas.

**FIGURE 1 Hydrogen Generator Diagram**



**FIGURE 2 Hydrogen Generator Application**





HYDROCARBONS-CANISTER CLEANING and SHIPPING  
Section III

LABORATORY RESPONSIBILITIES

### Approval Sign-Off Sheet

I certify that I have read and approve of the contents of this revision of the "Hydrocarbon-Canister Cleaning and Shipping - QA Plan, Section III, Laboratory Responsibilities" with an effective date of October 27, 2010. **Sign, date and return to the Ambient Monitoring Section Chief.**

Joette Steger, PPB Supervisor: Joette Steger 6/9/2011  
Donnie Redmond, Ambient Monitoring Section Chief: Donnie Redmond 6/30/11  
Carlton Blakley, Environmental Chemist: Carlton Blakley 10/27/2010  
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### **2.18.3 Hydrocarbons – Canister Cleaning and Shipping QA Procedure, Laboratory Responsibilities**

**Note:** The following is a list of "significant changes" from Revision 1.0.

1) QA updated per QAP/SOP 2.39 "Standard Operating Procedure (SOP) for Preparing Quality Assurance Plans/SOPs".

#### **2.18.3.6 CANISTER CLEANING**

##### **2.18.3.6.1 OBJECTIVES**

The canister cleaning procedure and equipment described in this section are recommended when obtaining integrated whole ambient air samples for subsequent analysis of VOC's. The cleaning procedure involves purging the canisters with cleaned humidified air and then subjecting them to high vacuum.

The purpose of canister cleaning is to insure that the canister interior surfaces are free of contaminants and that the canister meets a predetermined cleanliness (i.e. < 10ppbC NMOC). This level of cleanliness minimizes the potential for carryover of organic pollutants from one sample to the next, and helps ensure that the samples collected are representative.

The sample canister shipping described in this section is recommended when sending to the field cleaned, evacuated Summa passivated stainless steel canisters. The ambient air samples (non methane organic compounds (NMOC) hydrocarbons) are taken for subsequent analysis of VOC's.

##### **2.18.3.6.2 Canister Cleaning Equipment**

The equipment required to clean canisters includes a source of clean, humidified air to pressurize the canisters to a pressure of 30 psig, and a vacuum system for evacuating the canisters to < 0.5 Torr. Air from a standard oil-less air compressor will contain pollutants from the ambient air. In addition, various VOCs will be found in the compressed air because of the lubricants used in the air compressor. Hydrocarbon-free air may be purchased in cylinders and humidified before being used in the cleaning process. However, this approach may be cost-prohibitive. Figure 1 presents the schematic of a canister cleanup system that is suitable for cleaning up to 16 canisters concurrently. This, and any alternative system, must include a vacuum pump capable of evacuating the canisters to an absolute pressure of < 0.5 Torr.

The following equipment is incorporated in the canister cleaning system.

**AADCO Pure Air Generator** – An AADCO pure air generator is used to supply pure air for cleaning canisters. The pure air system consists of permeation driers and catalytic oxidizers that dry the air and oxidize hydrocarbon contaminants. The pure air generator uses air supplied by a "house" air compressor. Before entering the pure air system, the air passes through a coalescing filter to remove condensed moisture and hydrocarbon contaminants.

**Air cryotrap and purge valves** – The air cryotrap allows the cleaned air supply lines to be subjected to cryogenic temperatures to condense (1) water formed during the oxidation of hydrocarbons, (2) any remaining unoxidized hydrocarbons, and (3) other condensibles. Air cryotrap purge valves are used to purge these condensed components from the air cryotrap, as described in the opening procedure described below.

**Pressure regulators** – A high purity dual stage pressure regulator installed in each branch of the air supply line so that the maximum pressure attained during the cleanup procedure is controlled at 30 psig.

**Air humidifier** – The air humidifier shown in Figure 3 is an empty cartridge turned on its side. HPLC-grade water is placed in the cartridge prior to use. This process allows the humidification apparatus to supply cleaned, dried air that has been humidified to a relative humidity of ~ 80 %.

**Manifold air pressure valves** – Manifold air pressure valves used to isolate the air supply system from the manifold, or to make the pressurized air available to the manifold.

**Sixteen-port manifolds** – Sixteen-port manifolds designed to allow up to sixteen canisters at a time to be connected. Fewer canisters may be connected to the manifold if the vacant ports are sealed off with a plug.

**Turbo molecular vacuum pump** – A turbo molecular pump consisting of a roughing pump used to remove the moist cleaning air from the canisters while evacuating the canisters to about 100 mm Hg absolute and a high vacuum pump capable of reducing the pressure in the canisters to 5 mm Hg absolute.

**Vacuum cryotrap** – A U- shaped trap located in the vacuum manifold that is sized to fit inside a Dewar flask filled with cryogen. The purpose of this trap is to condense water vapor from the air that is pulled from the canisters during the vacuum cycle and prevent back-diffusion of organic vapors from the high-vacuum pump into the canisters during the vacuum cycle of the cleaning process.

**Vacuum source selector valve** – The vacuum source selector valve is a multiposition valve used to route either the roughing pump or the high vacuum pump to the eight-port manifold assemblies or isolate both pumps from the manifold assemblies.

**Compound absolute pressure gauge** – An absolute pressure gauge used to measure the pressure attained in the canisters during the vacuum and pressurization cycles of the cleaning procedure. The absolute pressure gauge must be able to measure absolute pressures from 40 psig down to 0.5 mm Hg absolute.

**Manifold valves** – The manifold vacuum valve and the manifold pressure valve are used to apply vacuum or pressure to the canisters, as required during the cleaning procedure.

**Manifold ports** – The manifold ports permit connection of the canisters to the manifold. Fittings that mate directly with the canister valve fittings are used.

These connections will not leak during the pressurization portion or the vacuum portion of the cleaning.



**FIGURE 1 Canister Cleaning Setup**



**FIGURE 2 Air Humidifier**

### **2.18.3.6.3 Canister Cleaning Procedure**

The cleanup system is prepared for use by checking the position of all valves. All valves should be closed initially, with the exception of the air bypass valve. Fill both the air source and vacuum pump vacuum flasks with cryogen and actuate the high-vacuum pump. Ensure that these vacuum flasks remain filled with cryogen throughout all cleanup activities. The inlet bellows valve is opened. Allow the system to stabilize for 10 minutes. After preparing the cleanup system, canister cleaning is performed using the following procedure.

1. Vent all canisters to the atmosphere by opening the green handled bellows valves on the canisters and log canister numbers in the Canister Cleaning Log Book. Fill in all pertinent data. The canister to be used for certification is determined by the last analysis.
2. Connect the canisters to be cleaned to the cleaning manifolds.
  - a. Securely tighten canisters to manifold with a 9/16 " or adjustable wrench. (Hold the valve handle on the canister valve while tightening the brass nut on the manifold to prevent the valve stem from turning).

- b. Open both the bellows valve on the canister and the associated valve on the manifold.
3. Remove collected moisture from the air cryotrap by opening and immediately closing the air cryotrap purge valves. Removal of the collected moisture should be performed at the beginning of each pressure cycle, so that the cryotrap does not plug with ice.
4. Begin the first vacuum cycle by actuating the roughing pump, opening the manifold valve and the vacuum controller gauge.
5. When the canisters are evacuated to approximately 100 mm Hg, as indicated by the absolute pressure gauge the turbo high vacuum pump should start automatically. The light should go from yellow to green.
6. Evacuate the canisters to  $< 0.5$  Torr absolute pressure (or less) and maintain the vacuum for 1 hour.
7. Close the manifold vacuum valve after the 1 hour high-vacuum period has been completed.
8. Begin the first pressure cycle by purging the air cryotrap (refer to Step 2) and then closing the air bypass valve.
9. Check the pressure regulators to verify that they are set to deliver a final pressure of 30 psig. Fill the canisters to 30 psig. As the final pressure is attained, the flow rates indicated on the air rotameters will drop to zero, regardless of the setting on the flow controllers because the pressure in the canisters and the pressure at the exit of the regulators reach equilibrium.
10. Close the manifold air pressure valve when filling is complete.
11. Release the pressure from the canisters after they have been under a 30-psig pressure for 5 minutes by opening the manifold pressure release valve.
12. Repeat steps 4, 5, 6, and 7 for Vacuum Cycle 2.
13. Repeat steps 8, 9, 10, and 11 for Pressure Cycle 2.
14. Repeat steps 4, 5, 6, and 7 for Vacuum Cycle 3, record final vacuum in the Canister Cleaning Log Book for all the samples except for the sample to be blanked.

#### 2.18.3.6.4 Canister Blanking Procedure

Prior to initial use, the cleanliness of all canisters should be assessed. After the initial blanking of 100% of the canisters, the blanking frequency can be reduced. One canister on a cleaning bank of sixteen canisters is considered representative and should be blanked. The selection of the canister to be blanked (from the bank of sixteen canisters) is determined by selecting the canister with the highest pre-cleanup TNMOC concentration on the manifold. This canister is selected because the potential for compound carryover is most likely to be the largest of any of the canisters on the manifold. The blank sample is analyzed using the analytical technique for ambient air samples. If this measurement meets the predetermined cleanliness criterion (i.e., <10 ppbC), then the other canisters on the manifold are considered clean. Blanking is a part of the overall canister cleanup procedure, and is described below.

1. Select the canister to be blanked by referencing the cleanup history logbook to determine the canister with the highest pre-cleanup TNMOC concentration.
2. Verify that all the canister bellows valves are closed. Disconnect the canister selected to be blanked.
3. Using the analytical technique, if the canister analysis meets the predetermined concentrations criterion (i.e. < 10 ppbC), then the blanked canister and all the other canisters on the bank of sixteen canisters are considered clean.
4. If the canister does not meet the cleanliness criterion (i.e. < 10 ppbC), it is reconnected to the manifold. The entire bank of canisters is given another vacuum and pressure cycle. After the additional cycle, the same canister is blanked again.
5. After the canister is blanked and has met the concentration acceptance criterion, it is reconnected to the manifold.
6. Assign a **Hydrocarbon Sampling Data Form** to the cleaned canisters, disconnect the canisters from the manifold, and place the canisters in the shipping containers..

Ref: EPA Technical Assistance Document for Sampling and Analysis of Ozone Precursors  
EPA/600-R-98/161 pages 98-107

#### 2.18.3.6.5 Operational Procedures

- a. Once the canister has been cleaned and evacuated, it is ready for shipment.

- b. If the canister has been sitting on the shelf for more than two days, verify the canister vacuum is still 28 in Hg or greater.
- c. Make sure shutoff valve is in the closed position.
- d. Attach the ¼ inch brass Swagelock cap to the shutoff valve and tighten finger tight. The brass caps are only tightened finger tight as their purpose is only to keep dust out the valve.
- e. Each canister should be observed for attached sample identification number.
- f. Each canister sample identification number is recorded on the " HYDROCARBON SAMPLING DATA FORM " and placed in the available shipping container.
- g. Attach shipping labels:(1) recipient and (2) return.



**FIGURE 3 High Vacuum Pump**



**FIGURE 4 Analytical System**

### HYDROCARBON SAMPLING DATA FORM

Site Name (two letter code) \_\_\_\_\_

Sample Collection Date: \_\_\_\_\_

Sample Collection Day (circle):

MON TUES WED THU FRI SAT SUN

Sample Collection Time: Start: \_\_\_\_\_ Stop: \_\_\_\_\_

Elapsed Time Meter: Start: \_\_\_\_\_ Stop: \_\_\_\_\_

(reset to 000.0 after run)

Sampler ID: \_\_\_\_\_

Ozone Analyzer Operating? Y / N

NO<sub>x</sub> Analyzer Operating? Y / N

Duplicate Sample for the Date: Y / N

Dup' Can #: \_\_\_\_\_

<b>Sample Canister #:</b> _____	
<b>Certification #:</b> _____	
	<u>Date</u> <u>Initials</u>
<b>Certification:</b>	_____
<b>Start Vac Lab:</b>	_____
<b>Start Vac Field:</b>	_____
<b>Return Pressure Field:</b>	_____
<b>Return Pressure Lab:</b>	_____
<b>Receiving Date:</b> _____	
<b>Analysis Date:</b> _____	

Comments: \_\_\_\_\_

\_\_\_\_\_  
\_\_\_\_\_

*THIS SECTION FOR LAB USE ONLY:*

Canister Condition: \_\_\_\_\_

Remarks on Sample Validity: \_\_\_\_\_

Highest Ozone Level \_\_\_\_\_ ppm; Site (if other than site where collected) \_\_\_\_\_.

NMOC Conc. ppbC \_\_\_\_\_ NMOC Analysis File # \_\_\_\_\_

Speciated Analysis File # \_\_\_\_\_

Comments: \_\_\_\_\_

\_\_\_\_\_

**Table 1 Summa Canister Logsheet**  
**SUMMA CANISTER LOGSHEET**

Canister ID No.	Site Location	Date Received	Date Analyzed	Analyzed NMOC	Cleaning Batch Number

\_\_\_\_\_  
Name

\_\_\_\_\_  
Date

**Table 2 UAM Summa Canister Cleaning Record**

UAM SUMMA CANISTER CLEANING RECORD

	Cans	Cleaned
Batch No	_____	_____
Operator	_____	_____
Start Date	_____	_____
End Date	_____	_____
Certification Can	_____	_____
Certification Date	_____	_____
Batch Release Date	_____	_____

Time	Pressure	
_____	_____	Vacuum Applied To Cans
_____	_____	Evacuate Cans to 0.5 Torr
_____	_____	Hold 1 Hour at < 0.5 Torr
_____	_____	Fill Cans with Humid Zero Air to 30 PSI
_____	_____	Cans Vented and Vacuum Applied to Cans
_____	_____	Evacuate Cans to 0.5 Torr
_____	_____	Hold 1 Hour at < 0.5 Torr
_____	_____	Fill Cans with Humid Zero Air to 30 PSI
_____	_____	Cans Vented and Vacuum Applied to Cans
_____	_____	Evacuate Cans to 0.5 Torr
_____	_____	Cans Clean; Close Valves and Remove

## HYDROCARBONS-SAMPLE ANALYSIS

### Section III

## LABORATORY RESPONSIBILITIES

## Approval Sign-Off Sheet

I certify that I have read and approve of the contents of this revision of the "Hydrocarbon-Sample Analysis - QA Plan, Section III, Laboratory Responsibilities" with an effective date of October 27, 2010. **Sign, date and return to the Ambient Monitoring Section Chief.**

Joette Steger, PPB Supervisor: Joette Steger 10/22/2011

Donnie Redmond, Ambient Monitoring Section Chief: Donnie Redmond 6/30/11

Carlton Blakley, Environmental Chemist: Carlton Blakley 10/27/2010

Chemistry Technician: Debra Lavery 11-3-2010

Chemistry Technician: \_\_\_\_\_

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### 2.18.3 Hydrocarbons – Sample Analysis QA Procedure, Laboratory Responsibilities

**Note:** The following is a list of "significant changes" from Revision 1.0.

- 1) QA updated per QAP/SOP 2.39 "Standard Operating Procedure (SOP) for Preparing Quality Assurance Plans/SOPs".

#### 2.18.3.8 SAMPLE ANALYSIS

##### 2.18.3.8.1 OBJECTIVES

The sample analysis described in this section is recommended when analyzing integrated whole ambient air samples of VOC's in Summa passivated stainless steel canisters. The VOC's are subsequently separated by gas chromatography (GC) and measured by multidetector techniques using **Method TO-12: Method for the Determination of Non-Methane Organic Compounds (NMOC) in Ambient Air Using Direct Flame Ionization Detection (FID)**.

##### 2.18.3.8.2 Sample Analysis Equipment

- 1) A Summa canister is an airtight, stainless steel container with an inner surface that has been electro polished and chemically deactivated. This process of chemical deactivation is the "Summa" process. DAQ uses 6 Liter summa canisters for ambient air samples and for collecting samples over time.
- 2) An analytical GC system is required, complete with a temperature-programmable oven, and either an integrator or PC-based data acquisition /analysis software. Also required are other accessories, including analytical columns and the gases required for GC-FID operation.

The GC system used is a Perkin Elmer Ozone Precursor System equipped with two direct flame ionization detectors (FIDs), ATD 400 air sampler interfaced with an IBM-compatible PC loaded with P E Nelson Turbochrom software.

In a flame ionization detector, the sample is injected into a hydrogen-rich flame where the organic vapors burn producing ionized molecular fragments. The resulting ion fragments are then collected and detected. The FID is nearly a universal detector. However, the detector response varies with the species of (functional group in) the organic compound in an oxygen atmosphere.

- 3) Chromatographic columns: (a) 50m x 0.32mm Al<sub>2</sub>O<sub>3</sub>/Na<sub>2</sub>SO<sub>4</sub> PLOT column and (b) 50m x 0.22mm 1- $\mu$ m BP-1 column.
- 4) Bubble flowmeter (mechanical or digital), capable of measuring accurate gas flow to 0.1 cc/min.

**2.18.3.8.3 Reagents**

- 1) Certified PAMS (Photochemical Assessment Monitoring Station) calibration mixture (Table 1) supplied from EPA.
- 2) Zero air, ultra pure air, used as sample blank in the Summa canister(s) for system blank(s) and oxidant for the FID.
- 3) Hydrogen, 99.99% or better, compressed or a generator as a fuel for the FID.
- 4) Helium, carrier gas for the chromatograph.
- 5) Ultra-pure water, HPLC grade or equivalent, for hydrocarbon (calibration mixture) stabilization in the Summa canisters.
- 6) Molecular sieve dryer for carrier gas.
- 7) Dry N<sub>2</sub> for purging the ATD cold trap chamber.

**2.18.3.8.4 Instrument Configuration and Parameters**

- a. P E Nelson Turbochrom data system configured to generate; a sample file, instrument file, process file, report file, and a sequence file for both channels.
- b. ATD 400 system parameters configured to carry the sample from the autosampler to the columns in the gas chromatograph system.
- c. AutoSystem gas chromatograph method set up to control ovens, ramp rates, injectors, detectors and timed events for the Deans' switch.

**2.18.3.8.5 Analysis**

- a. If the system has been shutdown for more than an hour or two, the system should be allowed to re-equilibrate by analyzing room air or zero air for a minimum of 12 hours.
- b. A system blank (in a Summa canister) is first run to evaluate the systems cleanliness. If the system blank contains more than 20 ppbC, discontinue sample analysis until the source of contamination is identified and removed.
- c. The system calibration (in a Summa canister) is then performed by analyzing the PAMS component standard. Before analyzing samples, ensure that the Deans' switch is properly set so the 1-hexene is on the Plot column and hexane is on the BP-1 column. Also confirm that all compounds in the standard are recovered at  $100 \pm 10\%$ .
- d. Another system blank is analyzed to evaluate the systems cleanliness and to check for carryover of the calibration sample. If the system blank indicates 3% or more carryover, discontinue sample analysis until the source of contamination is identified and removed.

e. Ambient air samples (in a Summa canister) are then analyzed. The resultant peaks are identified by retention times and quantified relative to the standard responses. A minimum of 10% of the samples is reanalyzed to determine precision. Ensure that duplicate samples are within  $\pm 10\%$ . If not reanalyze all of the samples in the batch.

f. The system calibration is then performed again by analyzing the PAMS component standard. Confirm that the recovery of each component in the standard is  $100 \pm 10\%$ . If not reanalyze all the samples.

g. An entry in the logbook is needed when sample analysis is successful.

#### 2.18.3.8.6 Quality Control

a. **Blank-** A laboratory blank is a canister of zero air that has been humidified with HPLC grade water. This blank must be analyzed before any standard or sample is run and when the system is conditioning.

b. **Calibration-** Analysis of the PAMS calibration mixture (Table 1). This calibration mixture must be analyzed daily before and after all samples, after any column change, and when major maintenance is performed.

c. **Precision** – 10% of all samples should be reanalyzed to determine precision.

#### 2.18.3.8.7 Preventive Maintenance

a. **Gas Pressures-** check pressures of the various gases and change as needed for the FID and carrier and cold trap purge. Gas cylinders should be changed when the cylinder pressure reaches 200 psi.

b. **Valves and fittings-** replace any leaking valves or fittings as needed.

c. **Data File Maintenance-** Periodically the Turbochrom data files should be transferred to a storage device (for example; tape drive or file server).

d. **Nafion Dryer** – replace annually.

#### 2.18.3.8.8 Trouble Shooting

a. **General Information-** See "Pamsgram Volume 18" for information pertaining to troubleshooting. It should be your first source of information for the hydrogen generator, gas chromatograph and Turbochrom. Space is provided in the instrument logbook for recording malfunctions, causes, fixes, and actions taken to prevent reoccurrences.

**Table 1 GC AutoSystem Method 2**

Time					
0.02	valve 3 off				
11.70	valve 3 on (this is determined by the elution time of 1-hexene and hexane)				
Temp1	45°	Time 1	15 min	Rate 1	5.0°/min
Temp 2	170°	Time 2	0 min	Rate 2	15.0°/min
Temp 3	200°	Time 3	6 min	Rate 3	End
Detector	250°				
Deans' Switch	0.20	valve 3 off			
	11.70	valve 3 on			

**Table 2 ATD 400 Method 1**

	<b>Set</b>	<b>Actual</b>		<b>Set</b>	<b>Actual</b>
Purge	1 min		Trap fast	yes	
Min psi	1		Cycle time	60	
Std inj	40.0	34.9	Trap low	-30	-30
Tube	1	1	Trap high	350	-29
Line temp	200		Trap hold	5.0	0.0
Oven temp	250		IN Split	no	
DSRB	1 min		OUT Split	3	
Valve temp	200	200	Transfer line	220	220
Inj / tube	99				

**Table 3 PAMS Calibration Mixture**

<b>Compound</b>	<b>Amount</b>	<b>Compound</b>	<b>Amount</b>
Ethylene	27.9	n-Heptane	26.2
Acetylene	32.6	Methylcyclohexane	32.1
Ethane	25.5	2,3,4-Trimethylpentane	26.1
Propylene	23.9	Toluene	40.7
Propane	26.7	2-Methylheptane	26.5
Isobutane	43.2	3-Methylheptane	26.7
Butene-1	25.5	n-Octane	31.5
n-Butane	27.3	Ethylbenzene	24.9
t-butene-2	43.2	m-Xylene	19.1
c-Butene-2	33.7	p-Xylene	21.1
Isopentane	43.6	Styrene	36.9
Pentene-1	26.0	o-Xylene	26.0
n-Pentane	25.5	n-Nonane	26.1
Isoprene	35.4	Isopropylbenzene	34.7
trans-2-Pentene	30.5	n-Propylbenzene	30.9
C-2-Pentene	37.7	m-Ethyltoluene	26.6
2,2-Dimethylbutane	42.4	p-Ethyltoluene	40.3
Cyclopentane	20.0	1,3,5-Trimethylbenzene	25.3
2,3-Dimethylbutane	55.1	o-Ethyltoluene	34.2
2-Methylpentane	22.4	1,2,4-Trimethylbenzene	41.2
3-Methylpentane	42.3	n-Decane	31.6
1-Hexene	61.6	1,2,3-Trimethylbenzene	25.6
n-Hexane	31.2	m-Diethylbenzene	41.2
Methylcyclopentane	27.1	p-Diethylbenzene	26.2
2,4-Dimethylpentane	42.5	Undecane	31.6
Benzene	31.3	Dodecane	37.9
Cyclohexane	42.6		
2-Methylhexane	81.9		
2,3-Dimethylpentane	<b>See Note 1</b>		
3-Methylhexane	26.9		
2,4,4-Trimethylpentane	31.5		

**Note 1 Unresolved pair - ratio at preparation 1:2 2-Methylhexane: 2,3-Dimethylpentane**

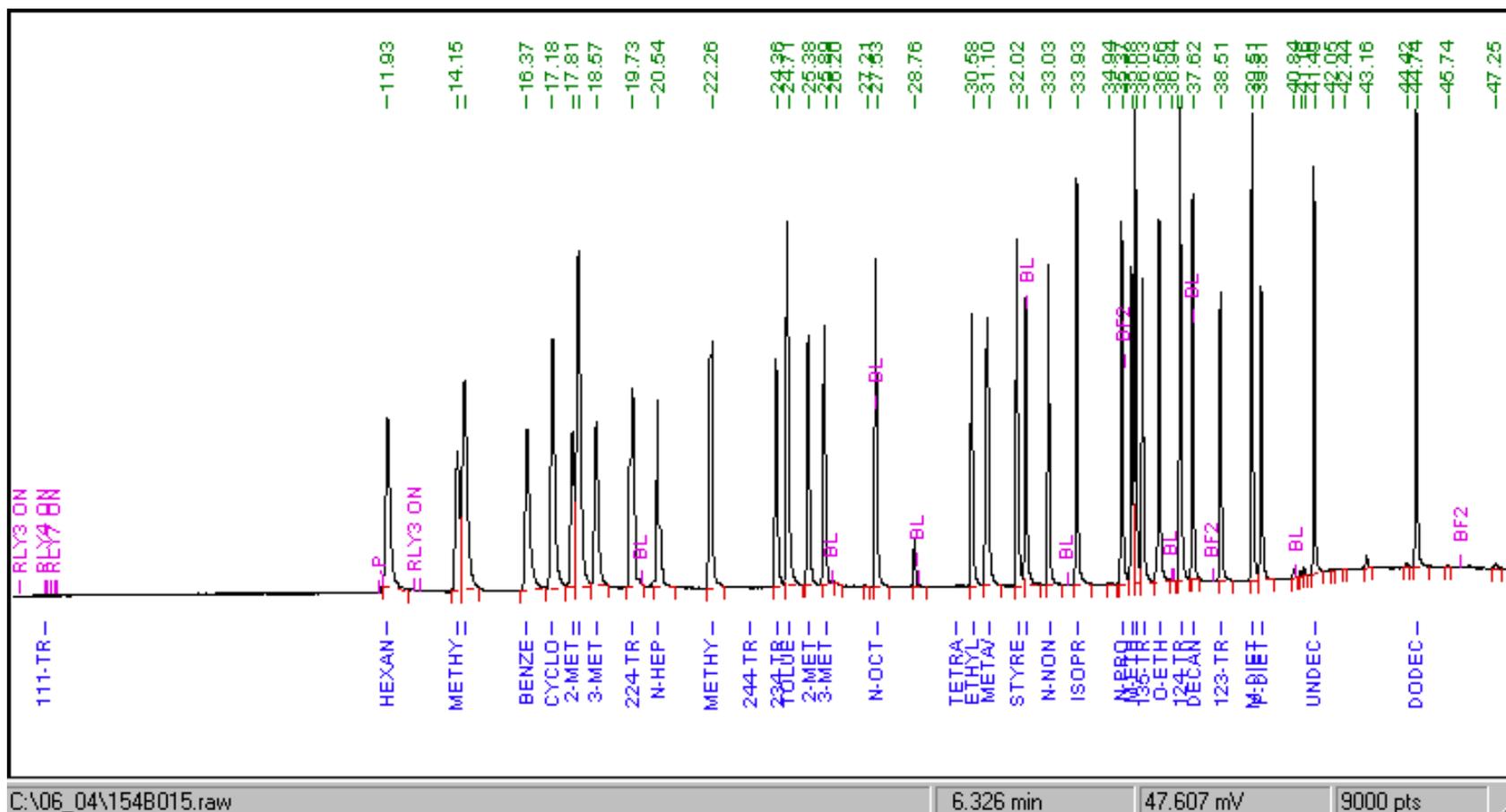


FIGURE 1 BP-1 Calibration Chromatogram

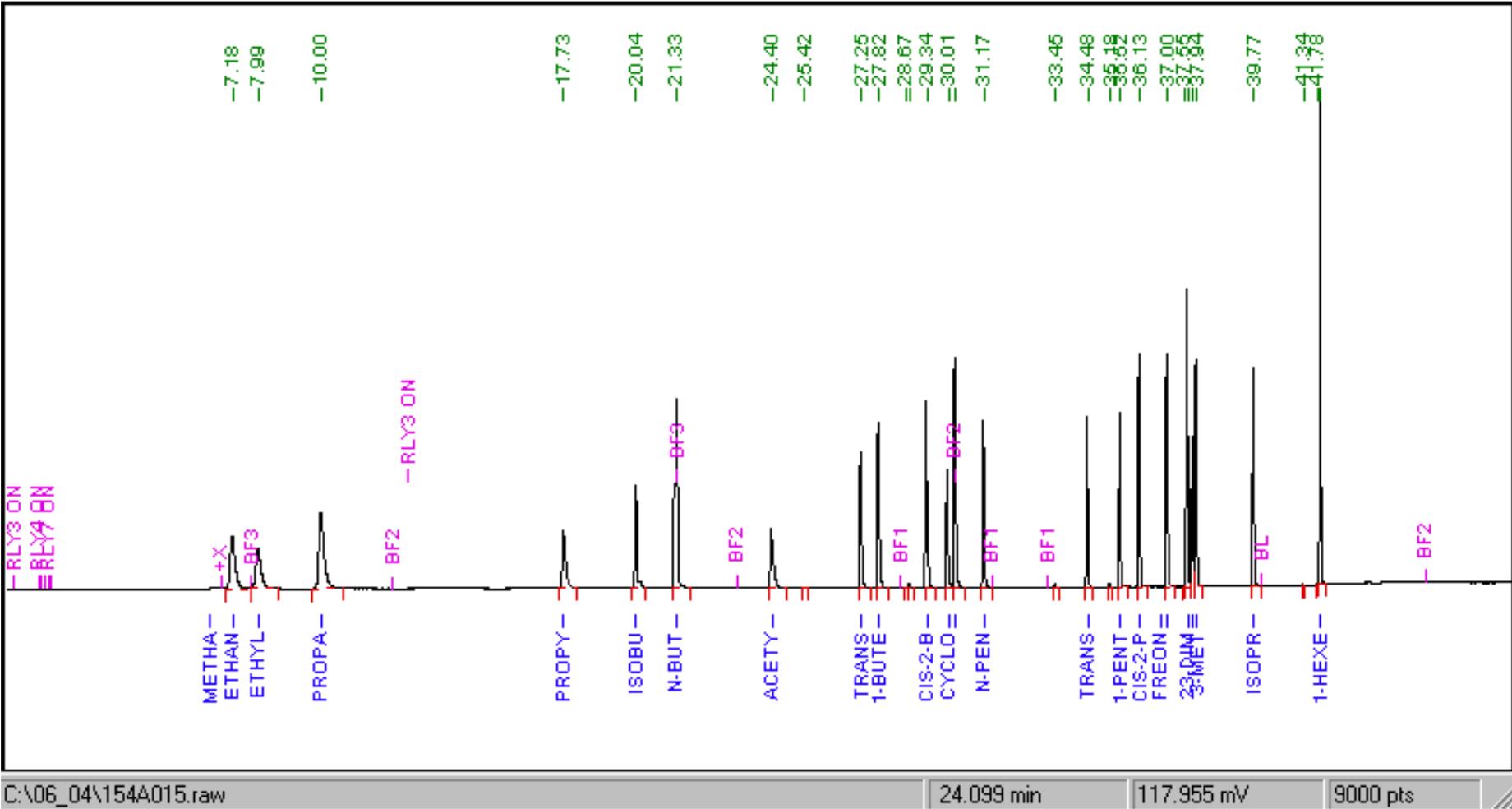


FIGURE 2 Plot Calibration Chromatogram

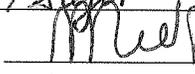
HYDROCARBONS-DATA REPORT  
Section III

LABORATORY RESPONSIBILITIES

## Approval Sign-Off Sheet

I certify that I have read and approve of the contents of this revision of the "Hydrocarbon-Data Report - QA Plan, Section III, Laboratory Responsibilities" with an effective date of October 27, 2010. **Sign, date and return to the Ambient Monitoring Section Chief.**

Joette Steger, PPB Supervisor:  6/22/2011

Donnie Redmond, Ambient Monitoring Section Chief:  6/30/11

Carlton Blakley, Environmental Chemist: Carlton Blakley 10/27/2010

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### 2.18.3 Hydrocarbons – Data Report QA Procedure, Laboratory Responsibilities

**Note:** The following is a list of "significant changes" from Revision 1.0.

- 1) QA updated per QAP/SOP 2.39 "Standard Operating Procedure (SOP) for Preparing Quality Assurance Plans/SOPs".

#### 2.18.3.9 DATA REPORT

##### 2.18.3.9.1 OBJECTIVES

The data reporting described in this section is recommended when reporting integrated whole ambient air samples of VOC's in Summa passivated stainless steel canisters. The VOC's are subsequently separated by gas chromatography (GC) and measured by multidetector techniques using **Method TO-12: Method for the Determination of Non-Methane Organic Compounds (NMOC) in Ambient Air Using Direct Flame Ionization Detection (FID)**.

##### 2.18.3.9.2 Hydrocarbon Data Report

Data reporting can be accomplished using instrument specific data collection software such as P. E. Nelson Turbochrom. Each chromatographic result is imported from a raw data file into Microsoft Excel and Microsoft Access where queries, tables, table relationships, forms and reports can be generated.

- 1) Queries select records from one or more tables in a database so they can be viewed, analyzed, and sorted on a common datasheet. The resulting collections of records are saved as a database object and can therefore be easily used in the future. The query will be updated whenever the original tables are updated. Types of queries are *select queries* that extract data from tables based on specified values, *find duplicate* queries that display records with duplicate values for one or more of the specified fields, and *find unmatched* queries display records from one table that do not have corresponding values in a second table.
  - a) Tables are grids that store information in a database similar to the way an Excel worksheet stores information in a workbook.
  - b) Table relationships are defined so that information can be used from more than one table at a time.
  - c) Forms are used as an alternative way to enter data into a database table.
  - d) Reports will organize and group the information in a table or query and provide a way to print the data in a database.

### 2.18.3.9.3 Data Quality Control/Assurance Procedures

#### Data Input – Standards and Raw Data

Procedure for inputting data into the database as of October 27, 2010.

1. open excel template (c:\mydocuments\Delta\_RT.XLS)
2. open file, select directory where files are located, select “\*.tx1” from the pull down menu in the file name field and hit "enter"
3. highlight desired files
4. click on "open"
5. check to see that "delimited" is chosen and then click on "next"
6. check comma box and then click on "finish"
7. repeat steps 5 and 6 for all selected files
8. select columns A through L and hit "control C" for the first text file to be transferred
9. Click on window, select the file "Delta\_RT.XLS", and click "OK"
10. If the file is not a QA file (samples), select the TEXT FILE spreadsheet, select edit, move or copy sheet, find spreadsheet, position the cursor in cell A1
11. Hit "control V"
12. If the file is a QA file (standards), select the QA TEXT FILE spreadsheet, select edit, move or copy sheet, find Delta\_RT.XLS; position the cursor in cell A1
13. Hit "control V"
14. click on window, select the text file just transferred, click "OK" close the file (click on the x or use file close); select no if it asks if you want to save data on the clipboard
15. Click on window, select the next text file to be transferred, click "OK"
16. Repeat steps 8 through 15, pasting the data into the next available spreadsheet (TEXT FILE 2, etc.)
17. When all of the data is transferred or all of the TEXT FILE spreadsheets are filled, select the file data spread sheet.
18. highlight row 2 through the last row containing data and hit "control C"
19. Click on window, select the file containing the database you are building, and click "OK"
20. Select the file data spreadsheet and position the cursor in column A of the first blank row
21. From the menu select edit and past special, check values, and click "OK"
22. Click on window and click on the file "Delta\_RT.XLS".
23. select the sample data spreadsheet
24. From the menu select edit, move or copy spreadsheet, select new book, check the box “create a copy”, and click "OK"
25. from the menu select tools, protection, unprotect sheet
26. highlight columns A through L and hit "control C"
27. From the menu select edit and paste special, check "values", and click "OK"
28. From the menu select data and sort, check on descending order, and click "OK"
29. highlight the second row and all of the following rows that contain data
30. hit "control C"
31. Click on window and click on the file containing the database you are building

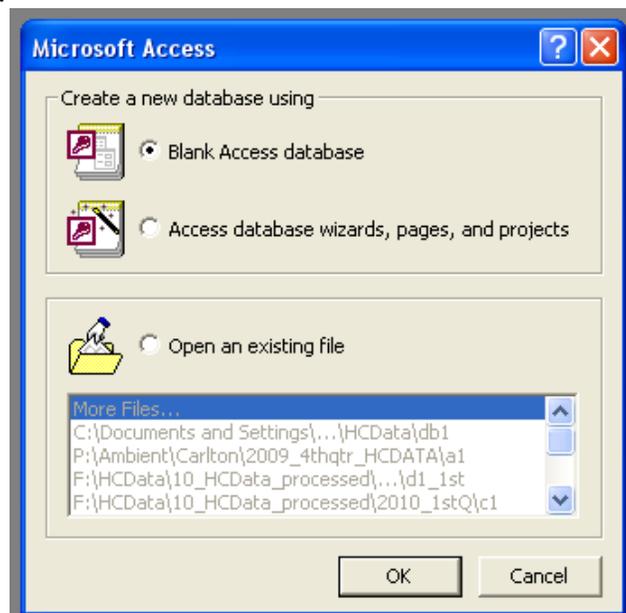
32. select the sample data spreadsheet, position the cursor in column A of the first blank row, and hit "**control V**"
33. Click on window and click on the file Delta\_RT.XLS
34. select the QA files spreadsheet, highlight row 2 down to the last row containing data, hit "**control C**"
35. Click on window and click on the file containing the database you are building
36. Select the QA data spreadsheet and position the cursor in column A of the first blank row
37. From the menu select edit and paste special, check "**values**", and click "**OK**"
38. Click on file and select save or save as, type a name in the file name (i.e., 02\_April) and click on save.
39. Click on window and select Delta\_RT.XLS
40. Select TEXT FILE 1
41. Hit delete and click on cell A1
42. Repeat steps 38 and 39 for each TEXT FILE spreadsheet until all of the data is erased
43. Repeat steps 1 through 40 for another batch of data until all data is transferred into the database

#### 2.18.3.9.4 Microsoft Access Database Quality Control Check

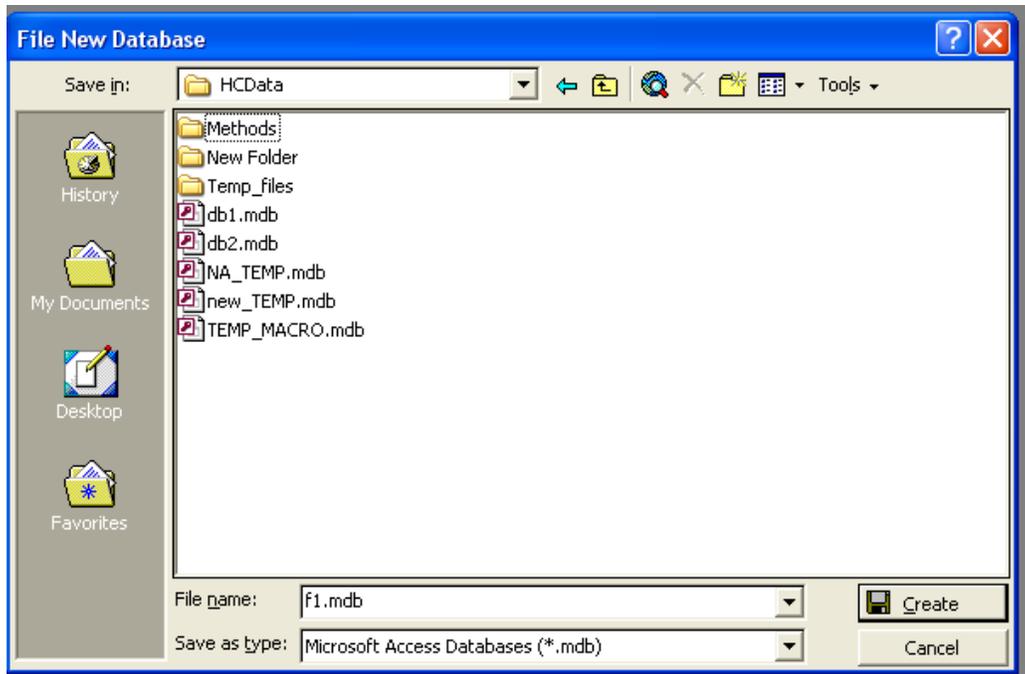
After creating the database perform the following actions:

"Copy" and "paste special (values)" data from spreadsheet the File Data, Sample Data and the QA Files to "**HCDData**" Excel spreadsheet.

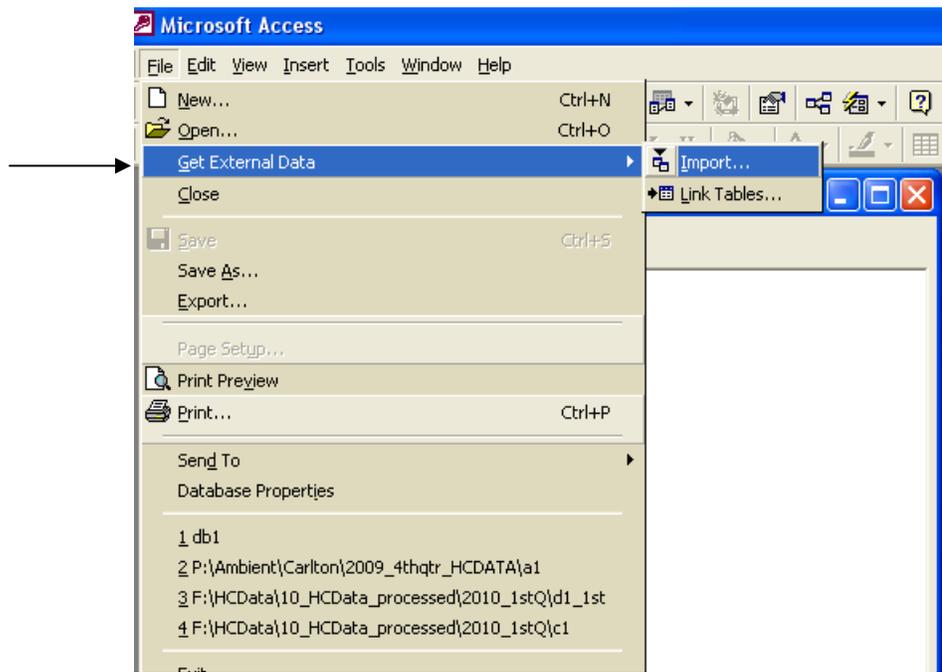
Create and name new blank access database using the "**Start**" button, click "**Blank Access database**", click "**OK**".



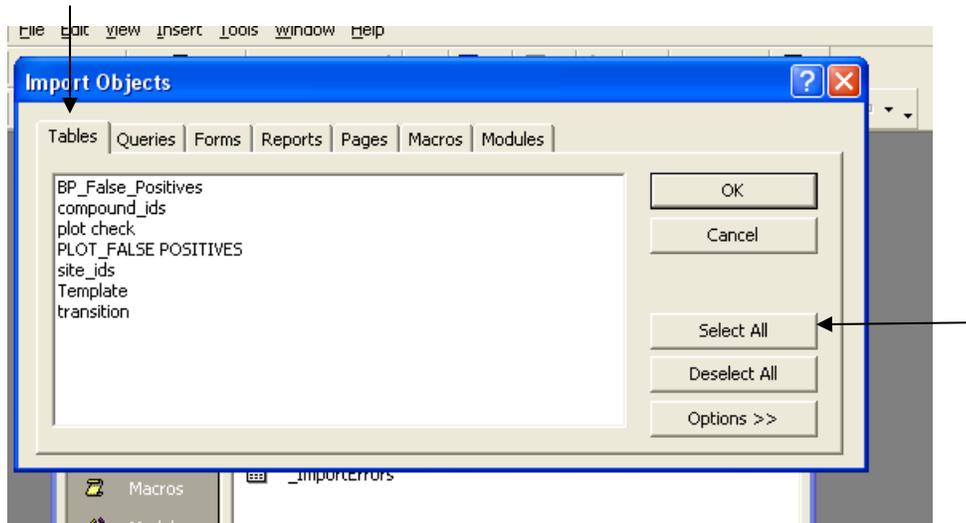
Name a new blank access database, save in folder where data is stored, click "**Create**".



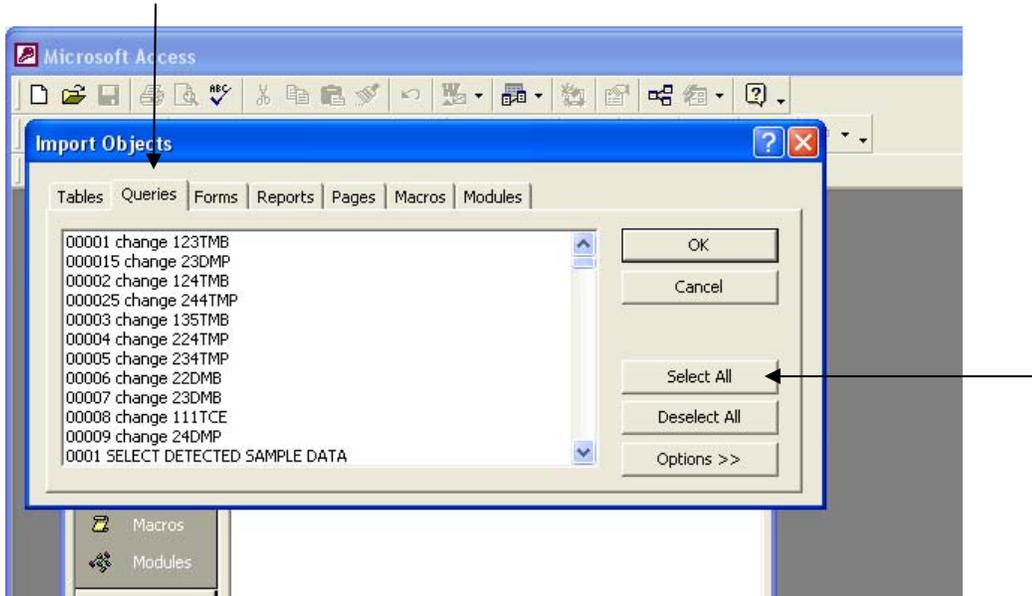
Open newly created database, under "**File**", select "**Get External Data**", select "**Import....**", Select a previous "template" database,



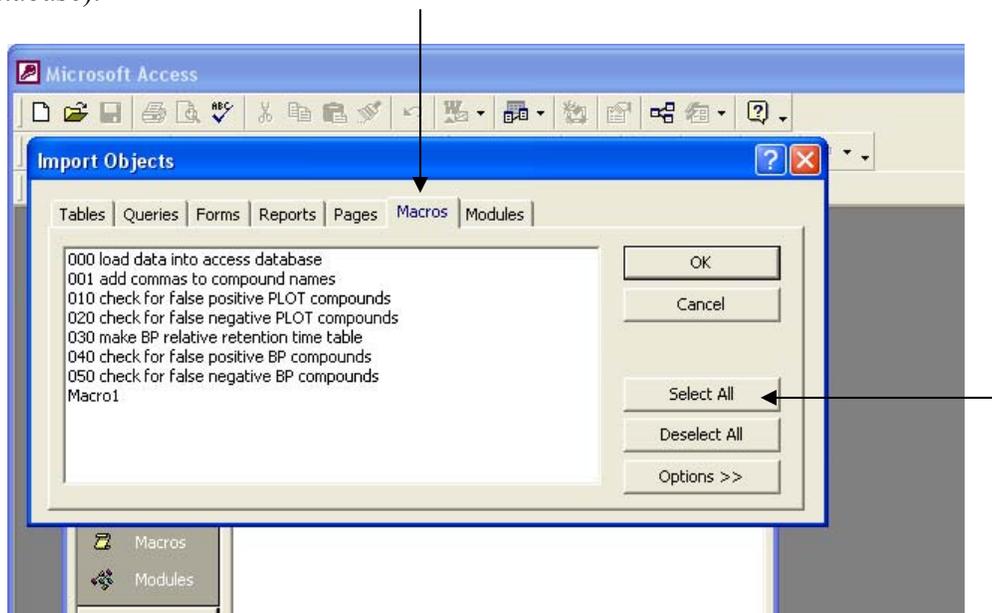
On "Tables" tab – click "Select All",



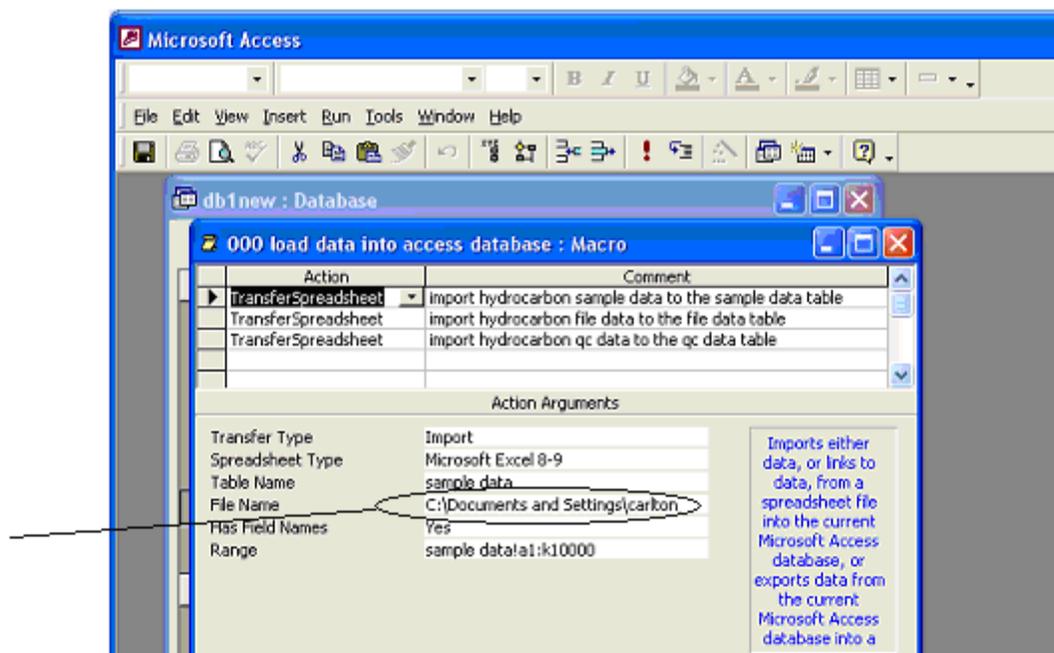
On "Queries" tab – click "Select All",



on "**Macros**" tab – click "**Select all**", hit "OK" (This transfers the table, queries and macros to the new database).



1. Run Macro 000 (load data from **HCDData** to the database).



**Note:** In design view, the Macro 000 **File Name** (in the Action Arguments section) for all three actions must have same location of the **HCDData** spreadsheet.

2. Run Macro 001 to add commas to compound names in the Sample Data Table.
3. Run query 0001 to select the sample data and paste it into the OUT Table.
4. Run query 001 to take the sample data in the OUT Table for the PLOT column and paste it into the PLOT\_DATA Table.
5. Run query 002 to take the sample data in the OUT Table for the BP-1 column and paste it into the BP\_DATA Table.
6. Run queries 110 through 125 to check for null compounds on the PLOT and BP-1 columns. If null compounds are detected, print out the table. To print the table, go to FILE and select Page Setup. In Page Setup choose Page and select Landscape. Return to Margins and set the right and left margins to 0 (the minimum value will be 0.25). Then select the print preview icon. If everything fits on one page, then select the print icon.
7. Run query 190 to make the PLOT Relative Retention Times Table.
8. Run query 195 to make the BP Relative Retention Times Table.
9. Run queries 200 to 202 to calculate the PLOT relative retention times (the relative retention times are calculated by subtracting the retention time of isopentane from the retention time of the compound; the values for isopentane in the PLOT Relative Retention Times Table are the retention times for isopentane).
10. Run queries 210 through 215 to check for false positives on the PLOT column. If false positive compounds are detected, print out the table. To print the table, select the print preview icon. If everything fits on one page, then select the print icon.
11. Run query 228 to make the Null Plot Compounds Table.
12. Run query 229 to calculate the relative retention times for the null PLOT compounds. Open the null plot compounds table in design view. Change field type for relative retention time to number.
13. Run queries 230 to 232 to check for false negatives on the PLOT column. If you get a "Data type mismatch in criteria expression." error, open the Null Plot Compounds Table in Design view and change the data type for the Relative Retention Time Field to "Number", the Field Size to "Double" and the Default Value to blank. If false negatives are detected, print out the table. To print the table, go to FILE and select Page Setup. In Page Setup choose Page and select Landscape. Return to Margins and set the right and left margins to 0 (the minimum value will be 0.25). Then select the print preview icon. If everything fits on one page, then select the print icon.
14. Run queries 240 through 244 to calculate the BP-1 relative retention times (the relative retention times are calculated by subtracting the retention time of toluene from the retention time of the compound; the values for toluene in the BP Relative Retention Times Table are the retention times for toluene).
15. Run queries 250 through 258 to check for false positives on the BP column. If false positive compounds are detected, print out the table. To print the table, select the print preview icon. If everything fits on one page, then select the print icon.
16. Run query 259 to make the Null BP Compounds Table.
17. Run query 260 to calculate the relative retention times for the null BP-1 compounds. . Open the null BP compounds table in design view. Change field type for relative retention time to number

18. Run queries 266 through 270 to check for false negatives on the BP-1 column. If false negatives are detected, print out the table. To print the table, go to FILE and select Page Setup. In Page Setup choose Page and select Landscape. Return to Margins and set the right and left margins to 0 (the minimum value will be 0.25). Then select the print preview icon. If everything fits on one page, then select the print icon.
19. Run query 2801 to make the PLOT\_AMOUNTS Table.
20. Open the PLOT\_AMOUNTS Table in Design view.
21. Edit the "Total Of Amount" Field Name by adding "PLOT\_" to the beginning of the name, i.e., change the Field Name from "Total of Amount" to "PLOT\_Total of Amount". This field contains the sum of all detected peaks (identified and unidentified).
22. Edit the "<" Field Name by adding "PLOT\_" to the beginning of the name, i.e., change the Field Name from "<" to "PLOT\_<". This field contains the sum of all unidentified detected peaks.
23. Run query 2802 to remove the "A" from the file name. This step and the two previous steps are necessary so that later the PLOT and BP tables can be combined.
24. Run queries 281 to 2835 to check for unusual relationships between PLOT compound amounts. If unusual relationships are detected, print out the table. To print the table, select the print preview icon. If everything fits on one page, then select the print icon.
25. Run query 2841 to make the BP\_AMOUNTS Table.
26. Open the BP\_AMOUNTS Table in Design view.
27. Edit the "Total Of Amount" Field Name by adding "BP\_" to the beginning of the name, i.e., change the Field Name from "Total of Amount" to "BP\_Total of Amount". This field contains the sum of all detected peaks (identified and unidentified).
28. Edit the "<" Field Name by adding "BP\_" to the beginning of the name, i.e., change the Field Name from "<" to "BP\_<". This field contains the sum of all unidentified detected peaks.
29. Run query 2842 to remove the "B" from the file name. This step and the two previous steps are necessary so that later the PLOT and BP tables can be combined.
30. Run queries 285, 286, and 288 through 290 to check for unusual relationships between various compound amounts. If unusual relationships are detected, print out the table. To print the table, select the print preview icon. If everything fits on one page, then select the print icon.
31. Run query 292 to check for outlier amounts on the PLOT column. If outlier amounts are detected, print out the table. To print the table, select the print preview icon. If everything fits on one page, then select the print icon.
32. Run query 294 to check for outlier amounts on the BP-1 column. If outlier amounts are detected, print out the table. To print the table, select the print preview icon. If everything fits on one page, then select the print icon.
33. Run queries 299 to 299c to check for peaks on the PLOT column, which are not integrated correctly. If incorrectly integrated peaks are detected, print out the table. To print the table, go to FILE and select Page Setup. In Page Setup choose Page and select Landscape. Return to Margins and set the right and left margins to 0 (the minimum value will be 0.25). Then select the print preview icon. If everything fits on one page, then select the print icon.

34. Run queries 299n through 299r to check for peaks on the BP-1 column, which are not integrated correctly. If incorrectly integrated peaks are detected, print out the table. To print the table, go to FILE and select Page Setup. In Page Setup choose Page and select Landscape. Return to Margins and set the right and left margins to 0 (the minimum value will be 0.25). Then select the print preview icon. If everything fits on one page, then select the print "icon".
35. Run query 600 to create county & site code table
36. Run query 6001 to clear transition table
37. Run query 60020 to remove blank rows from file data table
38. Run query 60021 to append file data table to transition table
39. Run query 60022 to add 43000 parameter to transition table
40. Run query 60024 to add 43102 parameter to transition table
41. Run query 601 to extract info from sample name to transition table
42. Run query 602 to check for Dec samples analyzed in Jan in transition table
43. Run query 603 to convert to EST
44. Run query 6031 to change format after 10am and DST
45. Run query 6032 to change format before 10am
46. Run query 6033 to change format before midnight
47. Run query 604 to create date in transition table
48. Run query 606 to add parameter code
49. Run query 607 to add site id
50. Run query 608 to make SF null for OPN season
51. Run query 609 to make SF R for OPN sites
52. Run query 624 to make sum of NMOC\_A
53. Run query 6241 to remove A from filename
54. Run query 625 to make sum of NMOC\_B
55. Run query 6251 to remove B from filename
56. Run query 6252 to append B to A
57. Run query 6253 to Sum A&B
58. Run query 626 to sum PAMS\_A
59. Run query 6261 to remove A from filename
60. Run query 627 to sum PAMS\_B
61. Run query 6271 to remove B from filename
62. Run query 6272 to append B to A
63. Run query 630 to add total NMOC
64. Run query 631 to add total PAMS
65. Run query 701 to flag low methane samples in transition table
66. Run query 701a update TYPE AQ
67. Run query 701b update TYPE AF
68. Run query 701c update TYPE AL
69. Run query 701d update TYPE AA
70. Run query 701e update TYPE AV
71. Run query 701f update TYPE AN
72. Run query 701g update TYPE 4

73. Run query 703 to flag bad methane samples in transition table
74. Run query 711 to make duplicates table
75. Run query 712 to make second duplicates table
76. Run query 713 to make second analysis results table
77. Run query 711 to delete duplicates table
78. Run query 714 to append first analysis of duplicate samples
79. Run query 715 to update amounts for duplicates table
80. Run query 808 to clear template table
81. Run query 809 to append template table
82. Run query 810 to Update template table w/ generic info
83. Run query 811 to Update template Millbrook
84. Close Access database