

**STANDARD OPERATING PROCEDURE
FOR
MEASUREMENT OF CARBONYL COMPOUNDS
FOR THE EPA SCHOOL AIR TOXICS PROGRAM**



**U.S. Environmental Protection Agency
Region 4, Science and Ecosystem Support Division
Athens, Georgia, 30605**

Acknowledgement

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Table of Contents

I. Introduction.....4

II. Installation.....5

 A. Sampler Siting.....5

 B. Sampler Installation.....6

III. Operating Procedure.....9

 A. Equipment and Supplies.....9

 B. Initial Receipt Activities.....10

 C. Setting the Flow Rate.....10

 D. Pump Calibration Factor Adjustment.....10

 E. Conducting the Sampling Event.....11

 Site Arrival Daily Activities.....11

 Preparing Sampler for a Sampling Event.....11

 Retrieving DNPH Cartridges.....13

 Field Data Recovery.....14

 Sample Shipping.....14

IV. Data Forms.....15

 ERG Carbonyl Compounds Sample Data..... 16

I. INTRODUCTION

This procedure is designed to provide instruction on collecting ambient carbonyl compounds (aldehydes and ketones) using the Buck Elite-5 personal air sampling pump. The procedure follows the tenets/specifications presented in EPA's Compendium Method TO-11A. Carbonyl compounds are adsorbed and converted into stable hydrazones using dinitrophenylhydrazine (DNPH) cartridges.

The Buck Elite is a battery-powered, data logging personal air sampler that is capable of programmed start/stop and duration sampling. The Buck Elite has a data logging feature that can store seven days of sampling data in its internal memory if data is logged every minute. The sampling time should always be 24 hours for the Toxics in Schools Study.

The Buck Elite was designed to operate from 0.8 standard liter per minute (800 cc per minute) to 6.0 standard liters per minute. The flow rate accuracy is guaranteed to $\pm 5\%$ of the calibration set point.

This SOP is designed to be a step by step method for operating the sampler to be used in conjunction with the manufacturer's operators manual. Laboratory Analysis Methodology may be referenced by contacting Eastern Research Group (ERG) directly at Julie.Swift@erg.com or Dave.Dayton@erg.com. Maintenance and troubleshooting should be conducted using the operator's manual.

FIGURE 1. Buck Elite-5 Sampling Pump



Photograph courtesy of A.P. Buck Inc.

II. INSTALLATION

A. Sampler Siting

Check the areas for safety. Ensure there will be enough room for the operator to move freely while working, and ensure physical conditions of the location will allow the operator to work safely.

The sampler should be set in a location unobstructed from any side. No tree limbs or other hanging obstructions should be above the sampler. It is suggested that the horizontal distance from the sampler to the closest vertical obstruction higher than the sampler be at least twice the height of the vertical obstruction.

Locate the sampler on a reasonably level structure with the probe inlet positioned at a height between two (2) and fifteen (15) meters above the ground.

B. Sampler Installation

The inlet of the probe must be at least 2 meters above ground level and the probe inlet line length can be no more than 2 feet long. Also, the Ozone (O₃) scrubber, sampling pump, and collection media (i.e., DNPH Cartridge) must be placed inside an enclosure/storage container to shield it from weather.

ERG has supplied an enclosure to meet the above requirements and is described below. The enclosure and collection components constitute a complete carbonyls sampling system with the exception of the sample pump and the system stand. The enclosure is comprised of 2 weather-tight NEMA 4 rated non-metallic units. Figure 2 below presents a view of the outside of the enclosure with its stainless steel (S.S.) inlet probe and supplied rain shield. The rain shield has been outfitted with a screen mesh to help reduce the potential for insects/debris being pulled into the sampler.

FIGURE 2. Carbonyl Sampling System Enclosure



Photograph courtesy of Dave Dayton, ERG

The recommended approach to constructing a stand and field mounting the enclosure is as follows:

1. Using the mounting tabs provided at each outside corner on the back panel of the enclosure chassis; attach the enclosure so that the top of the chassis is between 1.5 to 2 meters above ground level. If the enclosure will be attached to a wooden stand, 4 lag bolts and washers have been supplied for use. If the stand material is not compatible with applying lag bolts, hardware for attaching the enclosure will be the responsibility of the monitoring agency.
2. Remove the plug from the fitting positioned in the top left of the chassis. Securely fasten the S.S. inlet probe with rain shield to the fitting. With the probe assembly attached, the sample inlet will now be at the required height specification of at least 2 meters above ground level.
3. Each enclosure is outfitted with a hasp and a small padlock to provide some security. To open the enclosure, ensure that the hasp tongue is not over the hasp loop. Turn the screw fasteners located at the top and bottom of the front of the enclosure door counter-clockwise until they release. To close and seal the enclosure, fasten the hasp and turn the screw fasteners clock-wise until they set.

Inside the enclosures, the complete sampling train required to conduct carbonyl sample collection is pre-constructed. The O₃ scrubber is housed in the smaller of the two enclosures as presented in Figure 3. The O₃ scrubber is a temperature controlled/insulated denuder coated with potassium iodide (KI) and meets all of the specifications presented in the EPA NATTS TAD. Figure 4 presents a view of the inside of the enclosure with a DNPH cartridge and the Buck pump installed and Figure 5 shows detail of a DNPH cartridge.

The carbonyl sampling system employs an active temperature controller to maintain the O₃ scrubber at a predetermined set point. The temperature controller incorporates a power switch so it can be manually turned on and off as needed. For convenience, an unswitched AC power receptacle is provided to allow the Buck pump to remain plugged in and charging at all times. The calibrated flow meter has been mounted to a chassis wall to make it more convenient to use.

FIGURE 3. O₃ Scrubber

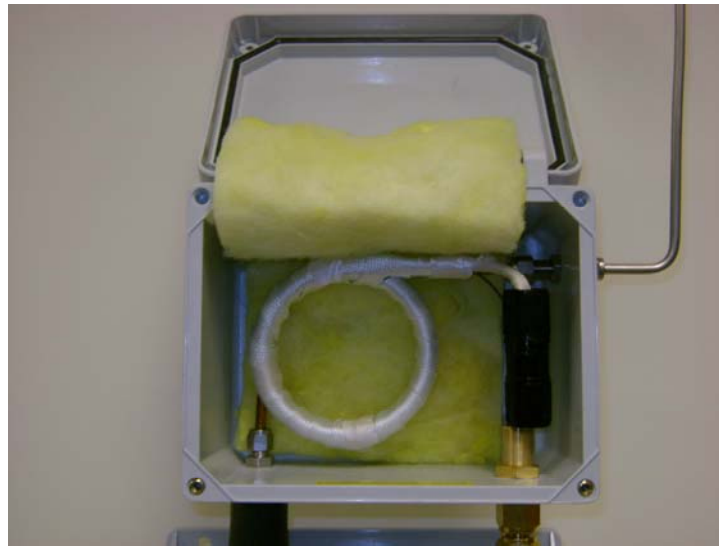


FIGURE 4. Inside View of Carbonyl Sampling System Enclosure

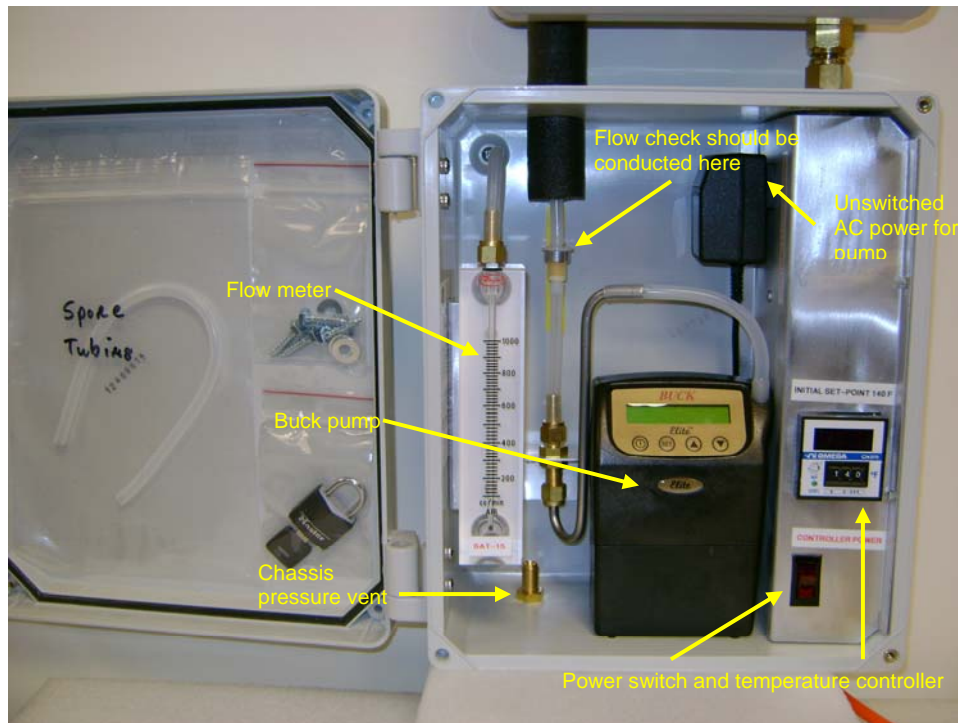
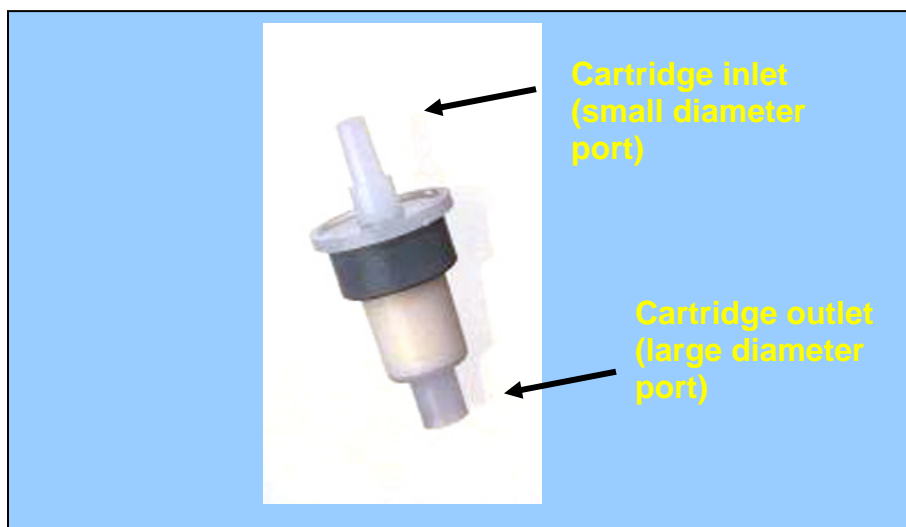


FIGURE 5. Waters DNPH Filter Cartridge



To complete assembly of the system:

1. Connect the length of platinum grade silicone tubing positioned at the exit of the DNPH cartridge holder (i.e., ¼" stainless tube) to the inlet of the sampling pump.
2. Insert the outlet (large diameter port) of the DNPH cartridge into the length of silicone tubing attached to the bottom of DNPH cartridge holder. Insert the inlet (small diameter port) of the DNPH cartridge into the length of silicone tubing attached to the top of the DNPH cartridge holder.
3. To perform pre- and post-event flow rate checks, remove the inlet of the DNPH cartridge from the silicone tubing and connect the flow meter per the instructions provided in the SOP (see Figure 4).
4. With the enclosure assembled and attached to the stand, level and secure the stand at the site. It is imperative that the enclosure be as close to perfectly level (on both the horizontal and vertical planes) to ensure that the flow meter provides an accurate flow measurement.

III. OPERATING PROCEDURE

A. Equipment and Supplies

Sampling system enclosures (received from ERG)
Ozone scrubber (received from ERG)
Platinum grade silicone tubing (received from ERG)

Calibrated flow meter (received from ERG)
Capped DNPH cartridge (received from ERG)
Foil shipping sleeves for sampled cartridges (received from ERG)
Chain-of-Custody (received from ERG)
Cooler with blue ice (received from ERG)
Buck Elite pump (supplied by EPA)
Tripod or stand
Logbook
Powderfree gloves

B. Initial Receipt Activities

1. Plug sampling system into AC power. Plug Buck pump charger into unswitched AC receptacle (provided inside enclosure). Charge the Buck pump internal battery continuously.
2. Check parts and components against the packing list.
3. Ensure that all electrical components of the sampling system will power up and are operational.

C. Setting the Flow Rate

1. Securely connect the flow meter to the DNPH cartridge inlet using the silicone tubing provided with the flow meter.

NOTE: The small diameter port on the DNPH cartridge is the inlet. The DNPH cartridges should be installed in the cartridge holder with the inlet in the "up" position.

2. Press the Menu key (Down arrow) to the Flow adjust menu.
3. Press and hold the SET key and use arrows to get desired flow (this clears all previous data). The design flow rate for carbonyl sampling for the study is 900cc/min.
4. Releasing the SET key will store the flow rate.
5. Press the ON/OFF key to return to the Main Display.

D. Pump Calibration Factor Adjustment

To adjust the pump calibration factor:

1. To measure the flow rate have the DNPH cartridge and flow meter connected as indicated in Figure 4.

2. Press the Main Display down arrow to the Calibration Mode.
3. Press the SET key and release, pump begins to flow at preset flow rate from the steps above in C. Setting the Flow Rate.
4. Observe the flow on the flow meter. Read the center of the ball in the flow meter at "eye level" for the best accuracy. It should match within +/- 4% of the 900cc/min design flow rate. Press and hold the SET key and adjust the Up or Down arrow to change the Factor for the pump speed to match the desired flow rate.

CALIBRATION Target = 0.9 Lpm (900 cc/min)

5. Press ON/OFF to return to Main Display
6. Record the data from the calibration in the field logbook.

E. Conducting the Sampling Event

Site Arrival Daily Activities

Record maintenance or collection activities in a logbook.

Preparing Sampler for a Sampling Event

1. Prepare sample paperwork. On the ERG Carbonyl Compounds Data Sheet, complete the "Lab Pre-Samp." and "Field Setup" sections. In the top part of the PAMS table, record the number under "Cartridge Lot #" and "Sample ID". Record any pertinent observations in the "Comments" section at the bottom of the form.
2. Make sure the flow rate is set and pump calibrated according to the instructions in Sec III B.
3. Inspect the DNPH cartridge to verify that end caps are in place and have not fallen off during shipping. If end caps are not in place, use a new cartridge.
4. Carefully install the outlet of the DNPH cartridge in the cartridge holder, making sure all connections are secure. Connect the flow meter to the inlet of the DNPH cartridge.
5. Press the down arrow key under the "EXIT" command. The "RUN/MENU" screen should now be displayed.

6. Turn the pump on using the up arrow key below the "RUN" command.
7. Record the indicated flow rate (from the flow meter) in the "PreSampling Flow Meter Reading" field on the ERG Carbonyl Compounds Data Sheet "Field Setup" section. Read the center of the ball in the flow meter at "eye level" for the best accuracy. ERG provided the flow meters calibrated and will determine the corrected flow from each flow meter's calibration curve upon receipt of the sample(s).
8. Turn pump off by pressing and holding the power button until the 5 second countdown ceases and power terminates.
9. Press the down arrow key under the "EXIT" command to return to the "RUN/MENU" screen.
10. Remove flow meter from the DNPH cartridge inlet and connect DNPH cartridge inlet to the top connecting tube of the cartridge holder.
11. Ensure that the temperature controller power is turned on and that the temperature set point is 140 °F (this is the initial set point and may be adjusted as required on a site specific basis). The temperature set point is input by rotating the 3 numerical thumb wheels located at the bottom of the controller.

NOTE: With a set point of 140 °F, the digital display will present an attained temperature that ranges from approximately 150 to 170 °F dependent on the cycle. This is normal.

12. Press the Menu key (Down arrow) to scroll down to reset mode. If previous sampling data needs to be erased then press and hold SET key and press Up arrow. Press Yes to clear all data. If previous sampling data does not need to be erased then use Down arrow to scroll down to Timing Routine menu.
13. By default Timing Routine is deactivated. To activate press and hold SET key and press Up arrow twice to get to Start/Stop screen.
14. To Set a Start Time press and hold SET key and push Down arrow, the top line displays the current time in the pump clock. Bottom line displays the start time for sampling. Set up a time of midnight for the given sample date for the pump to turn on unattended and start sampling.

15. Press and hold SET key to activate time change sequence and use Up or Down arrows to make changes for the parameter that is flashing. To advance to next parameter and make changes press and hold SET key to advance and use Up and Down arrows to make changes. To back up, press the ON key.
16. After Start time is set, press Down arrow to scroll to 5min/hr Cycle where pump can be setup up for 5min ON and 5 min OFF cycle within one hour block. By default all 5 minute segments are set to Y=ON. To change to N=OFF Press and Hold SET key and press UP or DOWN arrow. To advance to next segment Press and Hold SET key and use UP and DOWN arrow to make changes. If no changes are required press only the DOWN arrow to scroll to STOP Time set up screen. Enter the desired time for pump to stop sampling and shut down. If no Stop time is set pump will run until battery gets low.
17. Press only the Down arrow to scroll through all menus to activate other parameters like key lock, Log sample rate, Log Download or Clear, set time clock and back to the main screen where user can start sampling.
18. Press RUN key to start sampling, Pump will display Delayed Start Time. Then it will shut down within 30 secs and go into hold mode.

NOTE: If pump is turned on while it is in hold mode, user input will be required either to Cancel or Resume delayed Start Sampling. If clock time goes past programmed Start time, then pump will not turn on at programmed time. Delayed Start time run needs to be manually cancelled and start time needs to be setup again.

Retrieving DNPH Cartridges

1. Put on powderfree gloves.

NOTE: Gloves MUST be changed for each sample, (i.e. between retrieving a sample and preparing a new run) to prevent cross contamination.

2. Perform a post-sampling flow rate check. Disconnect the inlet port of the DNPH cartridge from the tube on the upper cartridge holder. Attach the flow meter to the inlet port of the DNPH cartridge.
3. Press the power button to activate the pump's display.
4. Press the down arrow key to exit the data screen.

5. Press the up arrow key (should be the button under the "RUN" command) to turn on the pump.
6. Record the indicated flow rate (from the flow meter) in the "Post Sampling Flow meter Reading" field on the ERG Carbonyl Compounds Data Sheet "Field Recovery" section. Read the center of the ball in the flow meter at "eye level" for the best accuracy. ERG has previously calibrated the flow meter and will determine the corrected flow from the flow meter's calibration curve upon receipt.
7. To turn pump off, press and hold the power button until the countdown ceases.
8. Remove the DNPH cartridge and immediately cap each end. Place the capped DNPH cartridge in the foil shipping sleeves, and write the sample day and the site ID on the outside of the sleeve.

NOTE: Do not use Sharpie to write the sample information on the outside of the shipping sleeves. Use ballpoint pen.
9. Place the cartridges in a polypropylene bag. Place the bag in a cooler maintained at approximately 4 °C using blue ice. Return the prepared cooler to ERG.

Field Data Recovery

1. Fill in the "Elapsed Time" information in the "Field Recovery" section of the ERG Carbonyl Compounds Sample Data Sheet.
2. Fill in the "Post Sampling Flow meter Reading" information in the "Field Recovery" section of the ERG Carbonyl Compounds Data Sheet.
3. Review the ERG Carbonyl Compounds Sample Data Sheet and make sure all blank spaces are filled.

Sample Shipping

1. Place the foil shipping sleeve containing the sample in a polypropylene ziplock bag along with the chain-of-custody. Seal the ziplock bag.
2. Place the sealed ziplock bag in the supplied cooler along with the blue ice.

NOTE: The samples are required to be shipped cold (approximately 4 °C).

3. On the pre-completed FedEx shipping deck, fill out the "Sender" section (i.e., with the sampling agency's address and phone number).
4. Send the cooler priority overnight to ERG.


NOTE: If the pre-completed shipping deck is lost, use the address below for shipping to ERG, and contact them directly for the associated FedEx account number.

Eastern Research Group, Inc.
601 Keystone Park Drive
Suite 700
Morrisville, NC 27560
919-468-7924

IV. DATA FORMS

All sample related run data forms will be supplied by ERG. Check the data sheets for completion after every setup or retrieval event. The operator is expected to keep a logbook to document all site activities, quality assurance activities, and sampling activities. The ERG Carbonyl Compounds Sample Data Sheet is presented below.

ERG Carbonyl Compounds Sample Data Sheet

		<div style="border: 1px solid black; padding: 2px; display: inline-block;">ERG Lab ID # _____</div>
CARBONYL COMPOUNDS DATA SHEET		
Lab Pre-Samp.	Site Code: _____ Collection Date: _____ City/State: _____ Cartridge Lot #: _____ AQS Code: _____ Duplicate Event (Y/N): _____	
Field Setup	Set-Up Date: _____ Operator: _____ Sys. #: _____ Pre-Sampling Rotameter Reading (cc/min): _____ Elapsed Timer Reset (Y/N): _____	
Field Recovery	Recovery Date: _____ Sample Duration (3 or 24 hr): _____ Post Sampling Rotameter Reading (cc/min): _____ Elapsed Time: _____ Cartridges Capped (Y/N): _____ Status: Valid Void (Circle one)	
Lab Recovery	Received by: _____ Date: _____ Refrigerator No: _____ Status: Valid Void (Circle one) Temperature: _____ If void, why: _____ Sample Volume (total Liters): _____	

PAMS	Sample Date	Sample Time	Sample Duration	Sample Volume	Cartridge Lot #	Sample ID	Lab ID	

Comments: _____
