

Roy Cooper  
*Governor*  
Elizabeth Biser  
*Secretary*  
Michael A. Abraczinskas  
*Director*



DAQ-09-002.2 Standard Operating Procedure (SOP)  
ATEC 8000 PAMS Carbonyl Sampler  
OPERATOR RESPONSIBILITIES

Revision 1



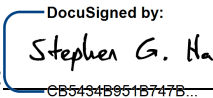
North Carolina Department of Environmental Quality | Division of Air Quality  
217 West Jones Street | 1641 Mail Service Center | Raleigh, North Carolina 27699-1641  
919.707.8400

1.0 Approval Sign Off-Sheet

I certify that I have read and approve of the contents of the ATEC 8000 PAMS Carbonyl Sampler Operator Responsibilities Standard Operating Procedure with an effective date of July 1st, 2021.

Director, Air Quality Division

Michael Abraczinskas

Signature:  Stephen G. Hall Acting for Michael Abraczinskas Date: 6/28/2021

Ambient Monitoring Section Chief

Patrick Butler

Signature:  Patrick Butler Date: 6/28/2021

Laboratory Analysis Branch Supervisor

Jim Bowyer, Environmental Program Supervisor II

Signature:  Jim Bowyer Date: 6/28/2021

Projects and Procedures Branch Supervisor

Joette Steger, Environmental Program Supervisor II

Signature:  Joette Steger Date: 6/28/2021

Primary SOP Author

Brian D. Velleco, Chemist I

Signature:  Brian D. Velleco Date: 6/28/2021

EPA Region 4

Designated Approving Official

Signature: \_\_\_\_\_ Date: \_\_\_\_\_

Disclaimer: This document, and any revision hereto, is intended solely as a reference guide to assist individuals in the operation of the instrument, related to the North Carolina Division of Air Quality's Ambient Monitoring Program

---

## SOP Acronym Glossary

---

ADQ - Audit of data quality

AQS - Air Quality System (EPA's Air database)

°C – degrees Celsius

CASTNET – Clean Air Status and Trends Network

CFR – Code of Federal Regulations

CGA – Compressed Gasses Association

Chief – Ambient Monitoring Section chief

COC – Chain of custody

DAQ - North Carolina Division of Air Quality

DAS – Data acquisition system

DEQ – North Carolina Department of Environmental Quality

DI – deionized water

Director – Division of Air Quality Director

DNPH – 2,4-dinitrophenylhydrazine

ECB – Electronics and Calibration Branch

e-log – electronic logbook

EPA – United States Environmental Protection Agency

FAS – fixed asset number

FB – field blank

FEM – Federal equivalent method

FRM – Federal reference method

hrs - hours

in – inches

IR - Infrared

L – liters

L/min – liters per minute

LAB – Laboratory Analysis Branch – DAQ Lab located at 4403 Reedy Creek Rd in Raleigh, NC

MDL – Method detection limit

MFC – mass flow controller

min – minute

---

mm – millimeter

NC – North Carolina

NIST – National institute of standards and technology

PAMS – Photochemical assessment monitoring station

PDF – portable document format

PM – Particulate matter

PPB – Projects and Procedures Branch

PPE – Personal Protective Equipment

psi – pounds per square inch

QA – Quality assurance

QA/QC - Quality assurance/quality control

QAPP - Quality assurance project plan

QC – Quality control

RCL – Reedy Creek Lab

RCO – Raleigh central office

SN – serial number

SOP - Standard operating procedure

TSA - Technical systems audit

UAT – Urban Air Toxics

USB – Universal Serial Bus

µg – microgram

VOC – Volatile organic compound

± - Plus or minus

## Table of Contents

---

1.0	Approval Sign Off-Sheet.....	2
	SOP Acronym Glossary .....	3
2.0	SCOPE AND PURPOSE .....	7
3.0	EQUIPMENT CHECKS AND MATERIALS .....	8
3.1	Equipment and Material List.....	8
3.2	Chemical and Material Checks.....	9
3.3	Support Equipment Checks.....	9
3.3.1	BGI-Tetracal Flow Meter Check .....	9
4.0	SITE CHECKS .....	9
5.0	DETAILED PROCEDURES.....	10
5.1	Preparing the ATEC 8000 for Sampling.....	10
5.2	Post-Sampling Pickup and Collection.....	17
6.0	DATA REVIEW.....	20
6.1	Self/Level 1 ATEC 8000 SIF and COC Review .....	20
6.2	Peer/Level 2 ATEC 8000 SIF and COC Review .....	20
6.3	Self/Level 1 Monitoring Site Logbook Review (Site Operator) .....	21
6.4	Peer/Level 2 Monitoring Site Logbook Review .....	21
7.0	FILE MANAGEMENT .....	21
7.1	Cartridge Chain of Custody and Sample Information Form Management .....	21
7.1.1	Gold Form (LAB).....	21
7.1.2	Pink Form (Site Operator).....	21
7.1.3	White and Yellow Forms (Site Operator/LAB) .....	22
7.2	Monitoring Site Logbook (Site Operator) .....	22
7.3	Monitoring Site Logbook (Peer/Level 2 Reviewer) .....	22
8.0	DATA QUALITY ASSURANCE AND DATA HANDLING .....	23
8.1	Records Management.....	23
8.2	Data Archival and Digitization.....	23
9.0	TROUBLESHOOTING AND CORRECTIVE ACTIONS.....	23
9.1	Blank ATEC 8000 Display Screen.....	23
9.2	Failing Leak Check (Leak Rate is Too High) .....	23
9.3	ATEC Sample Event Status Flags .....	23
9.4	Make-Up Samples: How and When to Schedule .....	24
10.0	REVISION HISTORY .....	24

11.0	REFERENCES .....	26
12.0	APPENDICES .....	26
	Appendix A – Carbonyls Cartridge Chain of Custody (COC) Form.....	27
	Appendix B – Carbonyls Cartridge Sample Information Form (SIF) .....	28
	Appendix C – ATEC 8000 Monitoring Site Logbook Entry Template .....	29

## 2.0 SCOPE AND PURPOSE

The scope and purpose of this document is to describe the steps required to operate an ATEC 8000 Carbonyl Sampler. The ATEC 8000 Carbonyl Sampler is deployed at the North Carolina Photochemical Assessment Monitoring Station (PAMS). The PAMS is located at the Millbrook Site at 3801 Spring Forest Rd. in Raleigh, NC 27616. The Millbrook Site serves to monitor the Raleigh, NC metropolitan statistical area air shed.

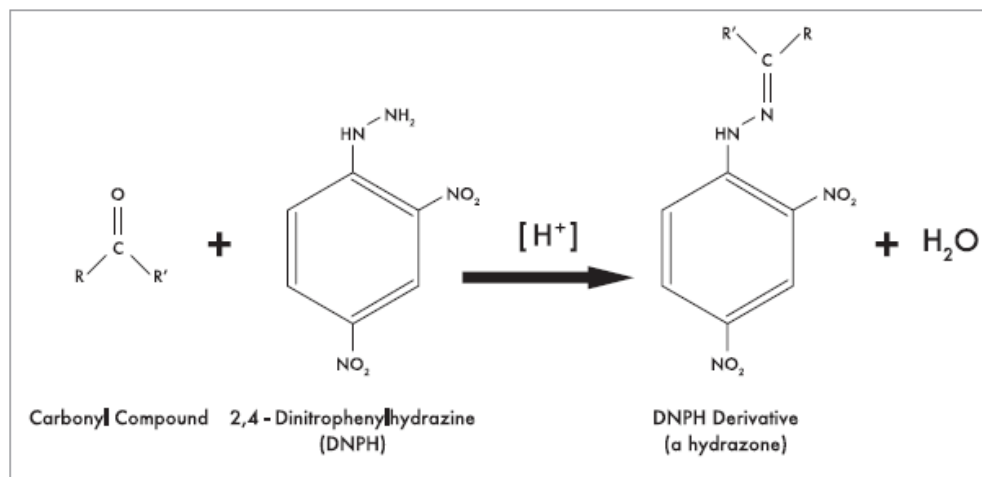


Figure 1: ATEC 8000 Cartridge Sampler



Figure 2: Waters Sep-Pak 2,4-Dinitrophenylhydrazine-Silica Cartridge

The carbonyls sampler collects ambient air through a 2,4-Dinitrophenylhydrazine-Silica (DNPH-Silica) impregnated cartridge as depicted in figure 2. The cartridges are installed into the ATEC 8000 and the sampler maintains a set flow with a calibrated mass flow controller (MFC) over three preset collection periods. The ATEC 8000 samples ambient air sequentially, in three 8-hour increments, through the cartridges to sample over a 24-hour period. The standard sampling periods are 04:00 – 12:00, 12:00-20:00 and 20:00-04:00. Carbonyls in the cartridge react with the 2,4-Dinitrophenylhydrazine over the collection period to form stable hydrazine derivatives via the following reaction mechanism:



*Figure 3: Carbonyl Derivatization Reaction Mechanism*

The collected samples over the 24-hour period can then be extracted and analyzed by **DAQ SOP-03.004.2 Thermo Ultimate 3000 Ultra-High Performance Liquid Chromatography Ultra-Violet and Mass Spectrometer Analysis**.

### 3.0 EQUIPMENT CHECKS AND MATERIALS

This section describes the equipment and materials that are required to complete the steps described in this document. Additional subsection(s) or standard operating procedures (SOPs) will also describe the equipment and materials as needed.

#### 3.1 Equipment and Material List

- ATEC 8000 Cartridge Sampler
  - Certified and installed via **SOP DAQ-09-002.1**
- ATEC Stylus Pen
- National Institute of Standards and Technology (NIST)-Traceable Flow Meter: BGI TetraCal with accessory pack or equivalent
- NIST-Traceable Clock: Fisherbrand Traceable Big-Digit Radio Atomic Wall Clock or equivalent
- NIST-Traceable Temperature Gun: Fisherbrand Traceable Infrared (IR) Thermometer Gun or equivalent
- Waters Sep-Pak 2,4-DNPH-Silica Cartridge or equivalent
- Cartridge Chain of Custody (COC) Form – An example form is in Appendix A
- Sample Information Form (SIF) – An example form is in Appendix B
- Personal Protective Equipment (PPE):
  - Nitrile Gloves
  - Non-Slip steel toed shoes or boots
  - Safety glasses: prescription or impact resistant are acceptable
- Portable Universal Serial Bus (USB) storage device (FAT32 format)



- Insulated cooler with ice packs
  - NIST-Traceable Thermometer with Time/Date, Min/Max Memory, or equivalent inside
- Freezer – to store cooler/ice packs [no samples]
- Temperature Logged Refrigerator for sample storage
- Clear Ziploc® bag or equivalent
- Monitoring Site Logbook (bound, numbered pages, laboratory grade)
- Scissors
- Clear Tape
- Pen (blue or black ink)

### 3.2 Chemical and Material Checks

---

Chemicals and materials used in this SOP are inspected prior to use and upon receipt at the Laboratory Analysis Branch (LAB). No damaged, uncalibrated, or expired equipment is to be employed in the discharge of procedures detailed in this document. Cartridges are lot checked for impurities as detailed in **DAQ SOP-03.004.2 Thermo Ultimate 3000 Ultra-High Performance Liquid Chromatography Ultra-Violet and Mass Spectrometer Analysis** prior to their use in this SOP.

### 3.3 Support Equipment Checks

---

#### 3.3.1 BGI-Tetracal Flow Meter Check

---

The BGI-Tetracal Flow Meter is calibrated annually by a third party NIST-traceable source. Ensure the calibration is within date before proceeding with the detailed procedures of this SOP. The expiration date can be determined by the adding one year to the sticker calibration date located on the bottom of the device.

## 4.0 SITE CHECKS

---

The site operator will confirm the following before proceeding with the detailed procedures within this document:

- **Site infrastructure check:** Is the shelter in good condition? Is there any damage or safety hazards to note? Is the electrical power on? Site HVAC running?
- **Sampler Check:** Is the ATEC 8000 powered on and pre-heated to ~50°C? Is the ATEC 8000 within the stickered certification date? Is the inlet connected to the rear of the sampler? If the sampler displays a blank screen, simply touch it to wake it up and populate the display. The temperature is shown in the bottom right corner of the main tab:

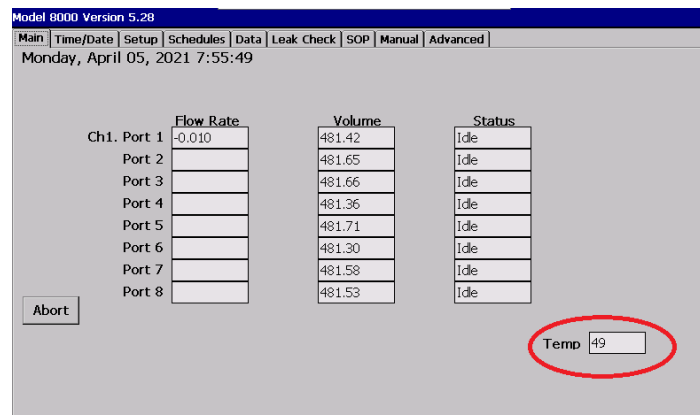
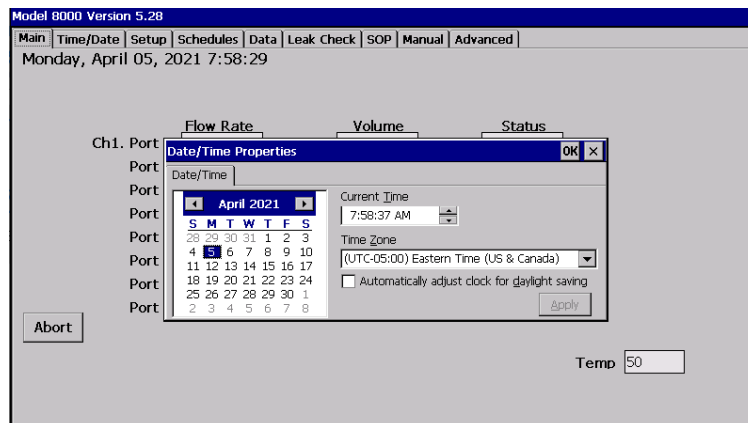


Figure 4: ATEC 8000 Main Screen with Oven Temperature Circled in Red

## 5.0 DETAILED PROCEDURES

### 5.1 Preparing the ATEC 8000 for Sampling

- 1 Obtain DNPH cartridges from the LAB with completed SIF and COC form. The **Laboratory Setup Information** section of each SIF must be completed by the LAB prior to use of the cartridges in the field. There will be one SIF form per cartridge.
- 2 Sign the chain of custody **Received by** field and fill in the **date/time** of sampling media acceptance.
- 3 Pack an insulated cooler with ice packs, the temperature min/max logger and sample cartridges for setup. Pack the BGI-Tetracal flow meter and accessories. **Note:** A best practice is to leave the temperature min/max device inside the cooler with ice packs and store it inside a freeze when not in use. Otherwise, the min/max functionality will need to be reset once the device has cooled to the temperature of the cooler
- 4 Drive to the Millbrook PAMS site. Bring supplies into the shelter and begin by completing the **Field Setup Information** section as detailed in the subsequent steps:
- 5 Locate the ATEC 8000 sampler serial number (SN) or Fixed Asset Number (FAS). Record the number in the **Sampler SN or FAS#** field on the Cartridge SIF form.
- 6 Record the sampler certification expiration date in the **Sampler Expiration Date** field on the SIF form.
- 7 Record the initials of the site operator, including the date and time of the cartridge setup in the **Site Operator (Initial, Date, and Time of Setup)** field of the SIF.
- 8 Record the date and time displayed on the ATEC 8000 sampler screen in the **As Found Sampler Display Date and Time** field on the Cartridge SIF form.
- 9 Record the date and time displayed on the NIST-traceable time keeping device along with the associated SN in the **NIST Device Date, Time, S/N** field on the SIF.
- 10 Calculate the absolute time difference, in minutes, and record the result in the **Time Difference (min)** field on the SIF. Circle the appropriate response in the **Time Difference Less than 5minutes?** field.
- 11 If the sampler display time differs more than or equal to 5 minutes, use the ATEC stylus to select the **Date/Time** tab on the sampler screen. A popup window titled **Date/Time Properties** will appear as shown below:



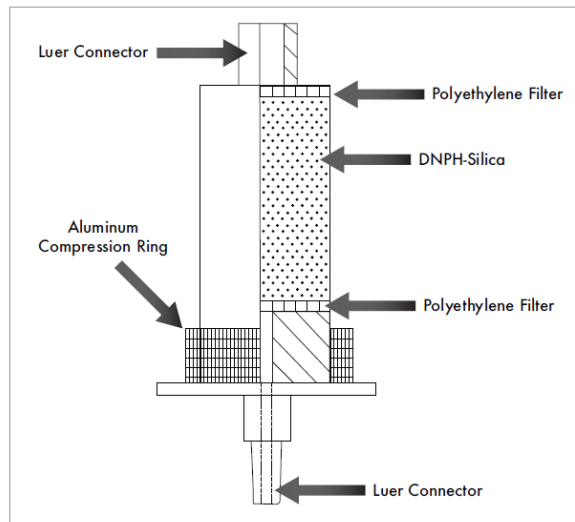
*Figure 5: ATEC 8000 Time/Date Tab*

- 12 Adjust the date and/or time to match the NIST-traceable date and time device. Click **Apply** when finished to push the settings to the instrument. Record the newly adjusted sampler display date and time in the **As Left Sampler Display Date and Time (if adjustment made)** field on the SIF form. If no adjustment was required, put a line through the entry field or write N/A. **Note:** the North Carolina Division of Air Quality (DAQ) does not observe daylight savings times on ambient monitoring equipment and no offset will be applied when daylight savings occurs. All recorded times are Eastern Standard Time.
- 13 Perform a pre-sampling flow check of the ATEC 8000 sampler by installing the designated flow check DNPH cartridge to the outlet line of port 1 on the ATEC 8000 sampler. Leave the cartridge inlet disconnected from the inlet line of port 1 on the ATEC 8000 sampler.



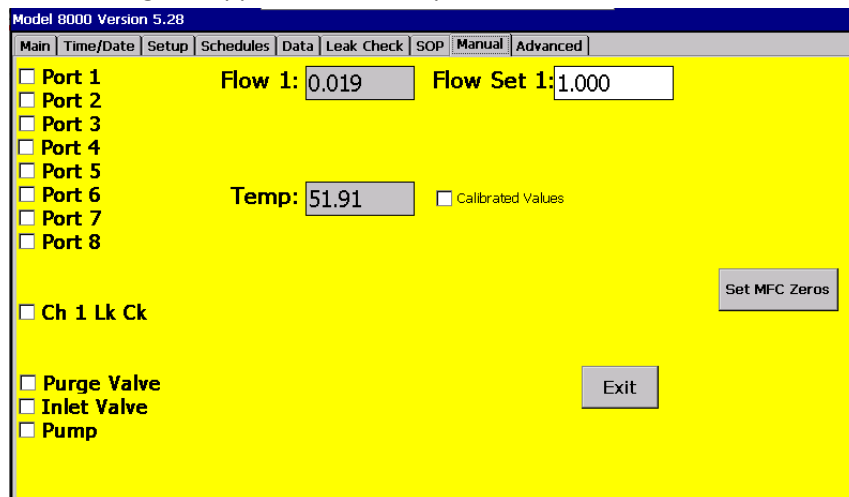
*Figure 6: ATEC 8000 DNPH Cartridge Flow Check Connection Setup*

The cartridge is inserted in this direction due to the design and a cutaway view is provided by Waters:



*Figure 7: Waters Cartridge Design*

- 14 Prepare to engage the sampler by selecting the **Manual** tab on the sampler display home screen with the ATEC Stylus Pen. The following will appear on the sampler screen:



*Figure 8: ATEC 8000 Manual Tab*

- 15 Turn on the sampler flow by selecting the **Manual** tab on the sampler display home screen with the ATEC Stylus Pen. Check the **Port 1** box, the **Pump** box, the **Inlet Valve** box, and check **Use Calibrated Values** box. The sampler will turn on and gas will begin flowing through the sampler. The MFC flow set point should be 1.000 L/min and the actual flow rate will be displayed in the **Flow 1** box on the sampler display screen as shown below:

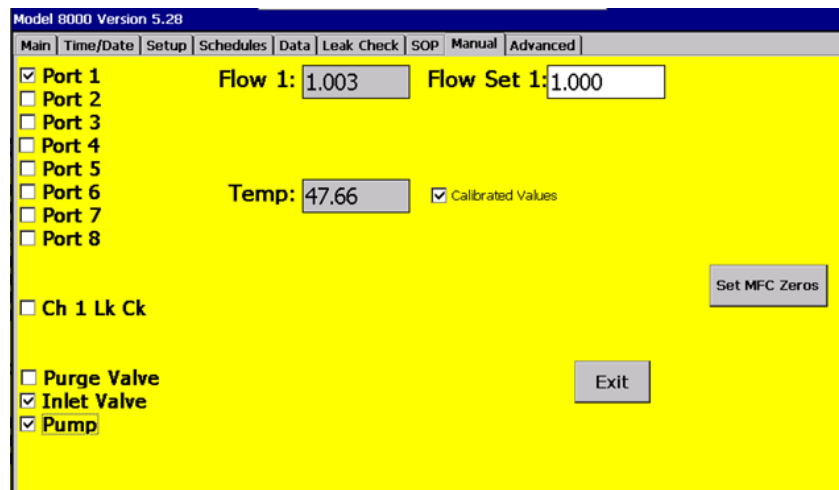


Figure 9: ATEC 8000 Flow Check Configuration for Port 1

Allow the sampler to equilibrate for 5 minutes.

- 16 Install the 0.1 to 1.2 L/min flow rate orifice into the BGI-TetraCal flow meter. Turn on the flow meter and allow it to zero and warm up for 30 seconds. During the warmup period, the flow meter will briefly display the flow range of the orifice that is inserted into the flow meter. Record the flow meter serial number and expiration date into the **NIST Flow Standard (SN, Exp. Date)** field on the SIF form.
- 17 Connect the BGI-TetraCal to the open end of the flow check DNPH cartridge installed on the sampler as illustrated below:

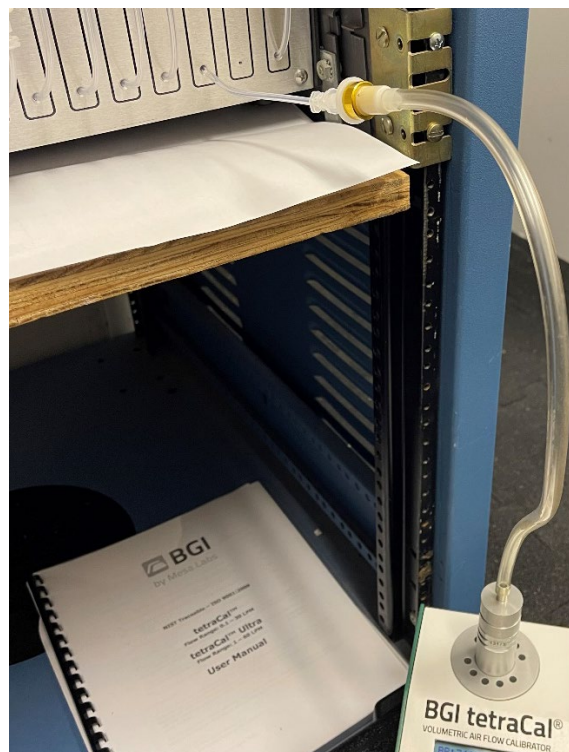


Figure 10: ATEC 8000 DNPH Cartridge Flow Check Setup

- 18 Allow the two systems to equilibrate for one minute and read the flow rate displayed on the BGI-Tetracal. Record the flow rate in the **Measured Pre-Sample Flow Rate (L/min)** field on the SIF form. If the measured flow rate falls outside the acceptable range (0.9 – 1.1 L/min) record the flow but also add a comment to the **Field Comments** field on the SIF form. An example comment for this situation would be **pre-sample sampler flow rate failure**. Proceed to the next step.
- 19 Turn off the BGI-Tetracal and disconnect it from the flow check DNPH cartridge.
- 20 Remove the flow check DNPH cartridge from the system.
- 21 Inspect the supplied DNPH cartridge from the LAB. It will be labeled with a sampler number on the foil pouch. This number should match the sampler number listed on the COC and SIF form. In the event these two do not match, the operator can correct the sample number on the foil pouch (or corresponding form in the event the SIF does not equal the COC) to match the sample number on the SIF and COC forms. The operator must document this change with a comment in the **Field Comments** section of the SIF form. An example comment would be **cartridge sample number did not match SIF/COC sample number; operator corrected**. These comments are paramount to reconstruct the sampling activities when post-collection review occurs.
- 22 Repeat steps 12-20 for port 2 and port 3.
- 23 Proceed to connect the regularly scheduled sample cartridge to port 1.
- 24 Connect the sample cartridge to the ATEC 8000 in the orientation shown below. Use a slight twisting motion while connecting the ATEC sampler to the DNPH cartridge to make luer fitting leak-tight connections. Be wary of twisting the ATEC inlet and outlet tubes as small tears are easily made and hard to see in the polyethylene tubing. A properly installed cartridge is shown in the following figure:



*Figure 11: ATEC 8000 DNPH Cartridge Collection Setup*

- 25 Select the **Leak Check** tab on the ATEC sampler touch screen. The following will appear:

*Figure 12: ATEC 8000 Leak Check Tab*

- 26 Press the **Start** button. The leak check takes approximately 20 seconds to complete.
- 27 Record the value in the **Flow Rate** box on the ATEC 8000 sampler screen in the **Leak Rate (L/min)** field on the SIF form. If the leak rate passes acceptance criteria, circle **Yes** in the **Leak Check Pass? (Circle one)** field on the SIF form. If the leak rate fails to meet criteria, circle **No** in the same field and see section 9.2 of this document for troubleshooting a failed leak check. Do not proceed with sample collection if the leak check fails.
- 28 Repeat steps 26-30 for port 2 and port 3. To perform a leak-check on the next port hit the next button in the lower left of the leak check tab. Then hit start. The top left corner of the display panel will indicate the current port being leak checked. A completed dialog status and leak check for Port 3 is shown below:

*Figure 13: ATEC 8000 Cartridge Leak Check Dialogue*

- 29 The scheduled sample date should be pre-recorded in the **Scheduled Sampling Date** field on the COC and SIF form. If that field happens to be empty, the sample date can be found coded in the **Sample #** field on the SIF

form. If the sample number is PC060121M1, the scheduled sample date is June 1<sup>st</sup>, 2021. Record the scheduled sampling date and time in the **DNPH Cartridge Scheduled Sampling Date/Time** field.

- 30 Select the **Schedules** tab on the ATEC 8000 display with the stylus. A new window will appear as shown below:

*Figure 14: ATEC 8000 Cartridge Scheduling Tab*

- 31 Use the drop-down arrow to select the Start Date for Port 1. This date should match the date recorded in the **DNPH Cartridge Scheduled Sampling Date/Time** field on the SIF form. The Start Time is always 04:00. Use the Left and Right arrows to adjust the start time, if needed. The Duration should always be 8.00 hours. Highlight the value entered in the Duration field. A new popup window will appear looking like a calculator; select 8.0, click apply when done. The Run Label should be the site name and port number: Millbrook PAMS 1.
- 32 Once the correct start date, time and duration has been entered, click the next button in the lower left corner of the Schedules screen. The same screen should be observed, but for port 2.
- 33 Use the drop-down arrow to select the Start Date for Port 2. Check the **follow previous port box** to immediately follow the previous sample port. This date should match the date recorded in the **DNPH Cartridge Scheduled Sampling Date/Time** field on the SIF form. The Start Time is always 12:00. Use the Left and Right arrows to adjust the start time, if needed. The Duration should always be 8.00 hours. Highlight the value entered in the Duration field. A new popup window will appear looking like a calculator; select 8.0, click apply when done. The Run Label should be the site name and port number: Millbrook PAMS 2.
- 34 Once the correct start date, time and duration has been entered, click the next button in the lower left corner of the Schedules screen. The same screen should be observed, but for port 3.
- 35 Use the drop-down arrow to select the Start Date for Port 3. Check the **follow previous port box** to immediately follow the previous sample port. This date should match the date recorded in the **DNPH Cartridge Scheduled Sampling Date/Time** field on the SIF form. The Start Time is always 20:00. Use the Left and Right arrows to adjust the start time, if needed. The Duration should always be 8.00 hours. Highlight the value entered in the Duration field. A new popup window will appear looking like a calculator; select 8.0, click apply when done. The Run Label should be the site name and port number: Millbrook PAMS 3.
- 36 Hit exit to apply the schedules and return to the main tab.



- 37 The **Status** field on the main screen should change to display **Waiting** as shown below. This may take a few seconds to appear. The **Waiting** status indicates the sampler has accepted the programmed start date, time and duration and will wait to start until the date and time has occurred locally on the unit.

Model 8000 Version 5.28

Main | Time/Date | Setup | Schedules | Data | Leak Check | SOP | Manual | Advanced

Monday, April 05, 2021 8:07:25

	Flow Rate	Volume	Status
Ch1. Port 1	-0.006	481.42	Waiting
Port 2		481.65	Waiting
Port 3		481.66	Waiting
Port 4		481.36	Idle
Port 5		481.71	Idle
Port 6		481.30	Idle
Port 7		481.58	Idle
Port 8		481.53	Idle

Abort

Temp 51

Figure 15: ATEC 8000 Status Screen

- 38 Sample setup for collection has now been completed. Follow the steps in Section 5.2 to retrieve the sampled DNPH cartridge within 48 hours after the sampling event completes.

## 5.2 Post-Sampling Pickup and Collection

- 1 Bring the insulated cooler with icepacks and NIST-Traceable Min/Max temperature device containing the next DNPH cartridge samples for setup and accompanying COC and SIF forms. Accept custody of the cartridges by signing the **Received By** field and fill in the **Date/Time** field. Bring the BGI-Tetracal and accessories to the site.
- 2 Upon arrival at the site, record your initials, date, and time of sample pickup in the **Site Operator (initial, date, time of pickup)** field in the **Field Recovery Information** section of the SIF form.
- 3 Touch the ATEC display screen to wake it up. The Status field on the main screen should display **Finished**.
- 4 Using the Stylus, select the **Data** tab and a screen like the example below should appear for the sampling event.

Model 8000 Version 5.32

Main | Time/Date | Setup | Schedules | Data | Leak Check | SOP | Manual | Advanced

Port 1

Started: 6/12/2021 4:00:02 AM

Stopped: 6/12/2021 12:00:31 PM

Flow: 1.00 1.00 1.00 lpm

Avg Min Max

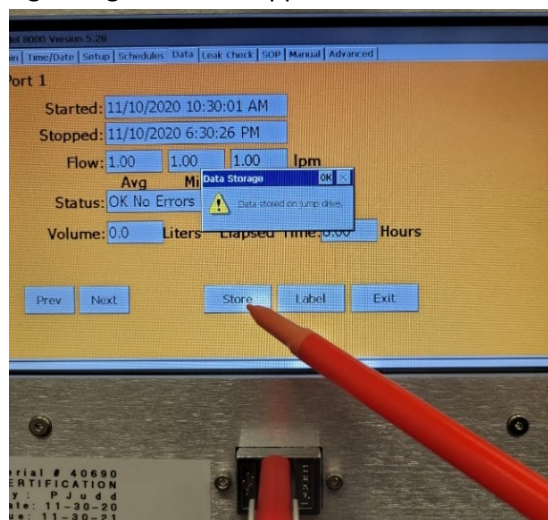
Status: OK No Errors

Volume: 481.5 Liters Elapsed Time: 8.00 Hours

Prev Next Store Label Exit

Figure 16: ATEC 8000 Data Screen Displaying Post-Sampling Data

- 5 Record the **Started** and **Stopped** dates and times that are displayed in the **Sample Start Date & Time** and **Sample Stop Date & Time** fields on the SIF form.
- 6 Record the **Volume** displayed on the ATEC 8000 screen in the **SAMPLE VOLUME (L)** field on the SIF form.
- 7 Record the **Elapsed Time** displayed on the ATEC 8000 screen in the **Sample Duration (hrs)** field on the SIF. The elapsed time should be within  $8 \pm 0.25$  hours. Denote any deviation within the **Field Comments** section of the SIF. An example comment for a sample event under 7.75 hours would be: **Sample event was less than 7.75 hours.**
- 8 Insert a USB storage device into the ATEC 8000. The port is located on the front panel underneath the touch display screen. Press the store button with the stylus. This will store data from all eight ports, regardless of the amount utilized. The following dialogue box will appear to confirm the data download:



*Figure 17: ATEC 8000 Data Download Confirmation*

- 9 Record any irregular status language displayed on the ATEC 8000 screen in the **Field Comments** section of the SIF form. If the sampler status says anything but **OK No Errors**, record the language. Do not proceed to the next step until the sample data has been downloaded to a USB storage device.
- 10 Repeat steps 5-9 for each cartridge by hitting the next tab in the lower left corner of the display. The current port will be displayed in the top left corner of the display screen.
- 11 While wearing nitrile gloves, remove each sampled DNPH cartridge from the ATEC 8000 sampler one port at a time. Install the end caps and place the cartridge in the labeled foil pouch that it was removed from during sample setup. Seal the foil pouch closed.
- 12 Place the foil pouch inside a clear Ziploc bag and seal closed. Place the cartridge in the insulated cooler, in between the ice packs.
- 13 If a field blank (FB) cartridge has been assigned, fill out the **Field Setup Information** as done in section 5.1 until the leak check section. The **Measured Pre-Sample Flow Rate (L/min)** will be equal to the flow check conducted during sample set up. If no field blank has been assigned, skip to step 18.
- 14 While wearing nitrile gloves, setup the FB sample collection on the assigned port. Open the FB DNPH sample cartridge foil pouch and remove the cartridge. Disconnect the cartridge end caps and place them back into the foil pouch.

- 15 Install the FB cartridge onto the assign channel on the SIF. Execute a leak check on the assigned FB port. Record the results in the **Leak Check Pass?** and **Leak Rate** field of the SIF.
- 16 Wait 5 to 10 minutes. Record the sample start and sample end time in the **sample start date & time** and **sample end date & time** in the **field recovery information** section of the SIF using the desired **Start** and **Stop** times from the NIST-Traceable Clock. Fill out the other applicable sections. The **Site Operator (Date, Time of Pickup)** information, the **Sample Volume (L)** which is equal to the volume of sample collected from assigned port from the current sample event and the **Sample Duration (hrs)** will be equivalent to the duration of the sample event collected for the assigned port for data reduction purposes. Record the results of steps 18-23 for the field blank cartridge as related to the port measured after completion.
- 17 When time has expired, remove the FB cartridge from the sampler, recap the cartridge and return it to the labeled foil pouch. Place the FB sample cartridge, SIF and COC form in their respective Ziploc bag and place the bags under ice packs in the insulated cooler.
- 18 Perform a post-sampling flow check of the ATEC 8000 sampler. Install the designated flow check DNPH cartridge to the outlet line of port 1 on the ATEC 8000 sampler. Leave the cartridge inlet disconnected from the inlet line of port 1 on the ATEC 8000 sampler as configured previously in Section 5.1, figure 6.
- 19 Turn on the sampler flow by selecting the **Manual** tab on the sampler display home screen with the ATEC Stylus Pen. Check the **Port 1** box, the **Pump** box, the **Inlet Valve**, and check **Use Calibrated Values** box. The sampler will turn on and gas will begin flowing through the sampler. The MFC flow set point should be 1.000 L/min and the actual flow rate will be displayed in the **Flow 1** box on the sampler display screen. Allow the sampler to equilibrate for 5 minutes.
- 20 Install the 0.1 to 1.2 L/min flow rate orifice into the BGI-Tetracal flow meter. Turn on the flow meter and allow it to zero and warm up for 30 seconds. During the warmup period, the flow meter will briefly display the flow range of the orifice that is inserted into the flow meter. Record the flow meter serial number and expiration date into the **NIST Flow Standard (SN / Exp. Date)** field on the SIF form.
- 21 Connect the BGI-Tetracal to the open end of flow check DNPH cartridge installed on the sampler as done previously in section 5.1, figure 10.
- 22 Allow the two systems to equilibrate for one minute and read the flow rate displayed on the BGI-Tetracal. Record the flow rate in the **Measured Post Sample Flow Rate (L/min)** field on the SIF form. If the measured flow rate falls outside the acceptable range (0.9 – 1.1 L/min) record the flow but also add a comment to the **Field Comments** field on the SIF form. An example comment for this situation would be **post-sample sampler flow rate failure**.
- 23 Repeat steps 13-17 for each additional sample port used: Port 2 and Port 3.
- 24 Turn off the BGI-Tetracal and disconnect it from the flow check DNPH cartridge.
- 25 Remove the flow check DNPH cartridge from the system.
- 26 At this point all fields in the **Laboratory Setup Information**; **Field Setup Information**; and **Field Recovery Information** of the SIF should be filled out in their entirety.
- 27 Remove the bottom copy (pink) of the SIF form and store at the PAMS site or in the site operator's office for future reference.

- 
- 28 Complete the remaining sections of the COC form. Fill in **SV** to denote transportation via state vehicle and put a line through or N/A in the **Shipment Tracking Number** field. Remove the bottom copy (pink) and store at the PAMS site or in the site operator's office for future reference.
  - 29 Place the SIF and COC form in the document sized Ziploc bag and seal it. Place the forms on top of the ice packs inside the insulated shipper containing the recently retrieved DNPH sample cartridge.
  - 30 **Please note:** If the next DNPH cartridge sample is being setup, the post sample flow rates recorded in steps 22-23 above can be recorded in the **Measured Pre-Sample Flow Rate (L/min)** field on each SIF form assigned to the next DNPH cartridge samples. Be sure to log all applicable details of the flow meter involved in the measurement as well as other steps detailed in section 5.1.
  - 31 Return the samples to the LAB.
  - 32 Upon arrival at the LAB, log in the samples collected. Measure the temperature of the collected samples with a NIST-Traceable IR gun. Mark the **Receipt Temperature** and **IR Gun Serial Number** on the SIF in the **Laboratory Receipt/Login Section**. Sign the **Sample Received By:** field and contemporaneously record the **Receipt Date/Time** on the SIF and COC. **Relinquish Custody** of the cartridges by signing the COC form. Store the samples in RCL fridge 2. Fill RCL fridge 2 in the **Post-sample Storage Location** on the COC. The LAB staff who extracts the collected cartridges will accept custody.
  - 33 Upload the downloaded data files from the ATEC 8000 to the P-drive in the following location: **P:\LAB (Laboratory Analysis Branch)\PAMS\ATEC 8000 Data**. Create a folder for each sampling event in the following formation: **YYYY-MM-DD**. An example data folder name of a sampling event performed on June 1<sup>st</sup>, 2021 would be: **2021-06-01**. Copy the files from the USB storage device to this newly created folder.
  - 34 The sample will then be extracted as detailed in **DAQ SOP-03.004.2 Thermo Ultimate 3000 Ultra-High Performance Liquid Chromatography Ultra-Violet and Mass Spectrometer Analysis** within 14 days of the end of collection.

## 6.0 DATA REVIEW

---

This section of the SOP describes steps required to perform a self/Level 1 review on the ATEC 8000 sampler data. Additionally, this section describes steps required for LAB personnel not directly involved in the ATEC 8000 operation process to perform a peer/Level 2 data review.

### 6.1 Self/Level 1 ATEC 8000 SIF and COC Review

---

Review the completed sections of the cartridge SIF and COC form. Review the SIF and COC form for completeness and accuracy. Confirm that all equipment SNs, expiration dates, times and other manual entries match the work performed. The LAB primary reviewer will review all entries under procedures in **DAQ SOP-03.004.2 Thermo Ultimate 3000 Ultra-High Performance Liquid Chromatography Ultra-Violet and Mass Spectrometer Analysis**.

### 6.2 Peer/Level 2 ATEC 8000 SIF and COC Review

---

---

Review the completed sections of the cartridge SIF and COC form. Review the SIF and COC form for completeness and accuracy. Confirm that all equipment SNs, expiration dates, times and other manual entries match the work performed. The LAB peer reviewer will review all entries under procedures in **DAQ SOP-03.004.2 Thermo Ultimate 3000 Ultra-High Performance Liquid Chromatography Ultra-Violet and Mass Spectrometer Analysis**.

### 6.3 Self/Level 1 Monitoring Site Logbook Review (Site Operator)

---

Review the completed sections of the logbook entry. Confirm that all equipment SNs, expiration dates, times and other manual entries match the work performed. Initial and date the logbook entry to indicate a level 1 review has occurred and submit for secondary, level 2 peer review. It is the responsibility of the primary, level 1, operator to find a suitable reviewer in a timely manner.

**Note:** The site logbook is to remain at the monitoring site. Logbook review will occur onsite. Regional or LAB staff not involved in the original collection of the sample can review this entry.

### 6.4 Peer/Level 2 Monitoring Site Logbook Review

---

Review the completed sections of the logbook entry. Confirm that all equipment SNs, expiration dates, times and other manual entries match the work performed. Data can be reviewed against the retained pink copy of the SIF. If there are any carbonless copy legibility issues, contact the LAB for a scanned copy of the original sheet for data review. Initial and date the logbook entry to indicate a level 2 review has occurred.

**Note:** The site logbook is to remain at the monitoring site. Logbook review will occur onsite periodically. LAB staff not involved in the original collection of the sample can review this entry.

## 7.0 FILE MANAGEMENT

---

This section of the SOP describes the different files generated during the ATEC 8000 operation processes and procedures as well as the individual required to manage the file. Files include the cartridge COC form, SIF, and monitoring site logbook.

### 7.1 Cartridge Chain of Custody and Sample Information Form Management

---

This form contains four-carbonless-copies (gold, pink, yellow, and white). During the DNPH cartridge sample setup and installation the COC and SIF form should only contain three of the four carbonless copies. The bottom copy should be pink.

#### 7.1.1 Gold Form (LAB)

The LAB keeps the gold copy as a record of DNPH cartridge SIF creation and COC form creation. If the bottom copy is gold, please remove the gold copy and return it to the LAB Chemist for record storage.

#### 7.1.2 Pink Form (Site Operator)

After the DNPH cartridge sample has been retrieved, all **Laboratory Setup Information, Field Setup Information, and Field Recovery Information** fields on the SIF form should be completed. Remove the pink copy of the SIF and COC form to keep as a backup record for the DNPH cartridge sampling event. Store the pink copy at the field site or in the site operator's office.

### 7.1.3 White and Yellow Forms (Site Operator/LAB)

Place the white and yellow copies of the form in the clear Ziploc bag and place in the insulated cooler for return to the LAB Chemist. These are used to transfer custody of the sample from the field to the LAB Chemist and to record the sampling data used in data reduction in **DAQ SOP-03-004.02**. These become part of the final data package and are electronically memorialized on the **P-drive**.

## 7.2 Monitoring Site Logbook (Site Operator)

---

This record is stored at the monitoring site and maintained by the site operator. During the sample setup steps, the site operator will fill out the logbook by initialing and dating the logbook entry using blue or black indelible ink. Include a summary of the site activities performed and results of instrument checks (if any performed). Routine site checks will be entered on the SIF form. The site logbook may be used as additional space if the Cartridge SIF form does not contain adequate space to record sampling information.

Site logbook entries should include:

- Cartridge Sample #
- Pre and Post Measured flow rate readings
- Sample Date
- Total Sample Volume
- Sample Duration
- Sampler status flags

A template site logbook entry is available in appendix C. If the template is to be used, cut, and paste it into the site logbook. Initial and date over the interface where the logbook entry meets the logbook page. Place a piece of clear tape over the entry. Secure the entire entry to the logbook.

Logbook entry mistakes/typos will be crossed out with a single line with the initials and date of when the correction was made. Write the corrected entry next to the crossed-out errata. The logbook entries are considered a secondary record of the sampling event. There may be instances when copies of the logbook entries must be sent via email or stored electronically for equipment troubleshooting and data validation purposes.

A printable version of the template site logbook entry is available at **P:\LAB (Laboratory Analysis Branch)\Forms and Templates**

## 7.3 Monitoring Site Logbook (Peer/Level 2 Reviewer)

---

The site operator will store the completed ATEC 8000 logbook entry within the Monitoring Site Logbook. The reviewer does not need to maintain this entry.

---

## 8.0 DATA QUALITY ASSURANCE AND DATA HANDLING

---

### 8.1 Records Management

---

Printed records will be held at the DAQ LAB for the period specified in the DAQ document retention guidelines.

### 8.2 Data Archival and Digitization

---

Reviewed COC records from each month will be scanned and archived on the P: drive. Scanned SIFs are digitized and memorialized on the P: drive per extraction batch.

---

## 9.0 TROUBLESHOOTING AND CORRECTIVE ACTIONS

---

The following troubleshooting and corrective actions can be performed by the site operator in the event a DNPH cartridge sampling issue occurs. The most common issues encountered during DNPH cartridge sampling are discussed below. Please refer to the instrument manual for additional troubleshooting steps in the event a sampling issue occurs that is not described below. Please contact the LAB electronics technician for further troubleshooting and corrective action steps.

### 9.1 Blank ATEC 8000 Display Screen

---

The ATEC 8000 display screen has a time out, or screen saver, feature that will cause the screen to go dark when not in use. Simply touch the screen to wake it up and you should be able to select sampler display menu screens and functions. If the screen does not come back on. Try to power the instrument on and off again. During power up the display screen should illuminate with various starting windows. If the display screen continues to remain blank, contact the LAB electronics technician for assistance.

### 9.2 Failing Leak Check (Leak Rate is Too High)

---

If a failing leak check occurs, where the leak rate exceeds the acceptance criteria listed in the SIF form, remove the sampled DNPH cartridge and re-install. Pay close attention to how snug the sampler line fittings attach to the DNPH cartridge. Remember the connection is not threaded; it is a luer-lock type fitting. This means that a slight push and twist motion should create a leak tight fitting between the DNPH cartridge and sampler lines. If the leak check continues to fail, the sampler may require maintenance. Contact the LAB electronics technician for further troubleshooting assistance. The polyethylene sample lines can be replaced in the field, this is the most likely point of a leak check failure. Notate any leak check failure and port location in the site logbook. Another sampling port can be utilized if the leak check passes. Comment on the SIF of the affected sample if this corrective action is executed.

### 9.3 ATEC Sample Event Status Flags

---

The ATEC sampler software performs several internal checks of the instrument performance during the sampling event. A successful sample event will display **OK No Errors** in the Status field as shown in Figure 16. Failure of these internal checks will produce a sampler status flag other than **OK No Errors**.

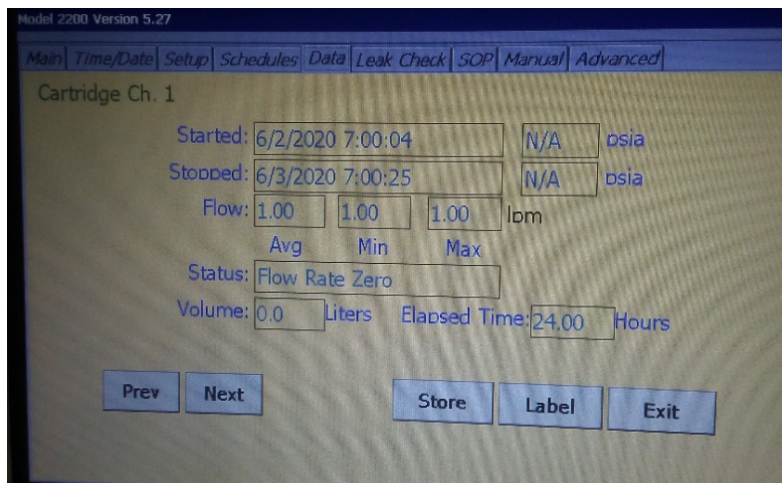


Figure 18: ATEC Data Download Example with an Error

In Figure 18 above, the sampler status is **Flow Rate Zero**. The sample event data stored by the sampler software should be downloaded to a USB storage device as detailed in the steps of this SOP. Email the LAB Electronics Technician and the LAB Chemist for further troubleshooting assistance.

#### 9.4 Make-Up Samples: How and When to Schedule

A make-up sample should be scheduled if the following occur:

- The site operator was unable to visit the site in a reasonable amount of time to set up the sample prior to an originally scheduled sample date. The site operator may schedule a make-up. Correct the sample number to match the make-up sample date. Please note the reason why the original sample was missed. The make-up sample must run from 04:00-12:00, 12:00-20:00, and 20:00-04:00 (three sequential 8-hour samples over 24 hours).
- The site operator returned to the site to retrieve the sample only to find the sampler did not run. The site operator must send the cartridges back to the LAB for processing and should request new cartridges from the LAB to be used for a make-up sample. The SIF form must contain any observations as to why this sample event was missed. E.g., power outage.
- PAMS sampling make-ups must be conducted within 14 days of the missed sample event and within the PAMS season of June 1<sup>st</sup> to August 31<sup>st</sup> annually. When possible, the make-up should occur before the next sampling event.

## 10.0 REVISION HISTORY

1. Rev 0 – **BDV** 04/15/2021 Original Publication
2. Rev 1 – **BDV** 06/16/2021 Changes:
  - a. Updated equipment and materials list
  - b. Section 3.2.1 – added how to determine the expiration date of the BGI TetraCal.
  - c. Added new PAMS Chain of Custody Form
  - d. Changed existing Chain of Custody (COC) Form to Sample Information Form (SIF) and changed previous references to the COC to SIF. The new COC is referenced where appropriate.



- 
- e. Update to field blank procedures – field blanks are to be collected on the prescribed sampling port per the approved PAMS QAPP upon sample pick up. Reworked sections 5.1 and 5.2 accordingly.
  - f. Formatted items in quotations to bold for emphasis.
  - g. Updated figures 6, 10 and 11 to reflect the cartridge orientation utilized by DAQ.
  - h. Updated figure 16 to reflect data generated from a sampling event, previous figure had zero in the volume field and might lead to confusion as this is not what should be displayed after a successful sampling event.
  - i. Section 5.2 – Added data upload location for clarification
  - j. Section 7.0 – Fixed typo in section header that referred to precision diluter process. Carryover from a previous SOP template.
  - k. Section 7.1 – Added SIF reference and simplified section to apply to the SIF and COC.
  - l. Section 8.2 – Updated data archival and digitalization to meet monthly data backup requirement
  - m. Section 9 – Removed any reference to “install a replacement ATEC 8000 sampler” as there is no back up sampler at this time. DAQ will attempt to procure one as budgetary funding allows.
  - n. Section 9.4 – Clarified make-up policy scheduling for PAMS.

---

## 11.0 REFERENCES

---

1. ATEC (2019) Model 8000 Automated Sampler: Operations and Maintenance Manual. Version 3.02. Malibu, CA. ATEC Atmospheric Technology.
2. Waters (2009) Waters SEP-PAK DNPH-Silica Cartridge: Care and Use Manual. Revision B. Milford, MA Waters Corporation.
3. United States Environmental Protection Agency: Office of Research and Development (1999). Compendium Method TO-11A: Determination of Formaldehyde in Ambient Air using Adsorbent Cartridge Followed by High Performance Liquid Chromatography (HPLC). Second Edition. Cincinnati, OH. William T. "Jerry" Winberry Jr., Silvestre Tejada, Bill Lonneman and Ted Kleindienst.
4. United States Environmental Protection Agency: Office of Air Quality Planning and Standards (2016). Technical Assistance Document for the National Air Toxics Trends Station Program. Revision 3. Research Triangle Park, NC. Authored by Battelle Memorial Institute (Columbus, OH).
5. United States Environmental Protection Agency: Office of Air Quality Planning and Standards (2019). Technical Assistance Document for Sampling and Analysis of Ozone Precursors for The Photochemical Assessment Monitoring Stations Program. Revision 2. Research Triangle Park, NC. Publication Number EPA-454/B-19-004.

---

## 12.0 APPENDICES

---

1. Appendix A – Carbonyls Cartridge Chain of Custody (COC) Form
2. Appendix B – Carbonyls Cartridge Sample Information Form (SIF)
3. Appendix C – ATEC 8000 Monitoring Site Logbook Entry Template

Appendix A – Carbonyls Cartridge Chain of Custody (COC) Form



DAQ-16-008 Carbonyl Cartridge Sample Chain of Custody Form

Sample ID	Pre-sample Storage Location	Sample Type	Scheduled Sample Date	Shipment Method*	Shipment Tracking Number	Post-sample Storage Location
Relinquished by	Date/Time		Received by		Date/Time	

DAQ-16-008  
 Revision 0.1  
 Effective: 07-01-2021

\*Shipment Abbreviations: **UPS** = United Parcel Service  
**FEDEX** = FedEx Corporation  
**SV** = State Vehicle

Appendix B – Carbonyls Cartridge Sample Information Form (SIF)

North Carolina Division of Air Quality Laboratory Analysis Branch DAQ-16-009 Carbonyl Cartridge Sample Information Form (SIF)										
<i>NC DAQ Laboratory Analysis Branch: 4403 Reedy Creek Road, Raleigh, NC 27607 Phone: (919)-733-9777</i>										
<i>Document #: DAQ-16-009, Revision 0.0, Effective: 7/1/2021</i>										
Laboratory Setup Information										
Sample Program (Circle one):      PAMS      UAT										
Sample Type (Circle one):    SAMPLE    FIELD BLANK    TRIP BLANK    DUPLICATE/COLLOCATED    SAMPLER CERTIFICATION										
QC Sample Type (Circle One):    CARTRIDGE BLANK    CARTRIDGE SPIKE    CARTRIDGE SPIKE DUPLICATE    MDL SPIKE    PT SPIKE    CATRIDGE LOT BLANK										
Sampling Site:					Sample #:					
DNPH Cartridge Lot#:					Sample Channel #:					
Lab setup by (initial & date):					Scheduled Sampling Date:					
Field Setup Information										
Sampler SN or FASH#:					Site Operator (initials, date & time of setup):					
Sampler Expiration Date:					NIST Device Date, Time and S/N:					
As Found Sampler Display Date & Time:					AS Left Sampler Display Date, Time: (if adjustment made)					
Time Difference (mins):	Time Difference less than 5 minutes?									
	Yes	No								
NIST Flow Standard (S/N and Exp. Date):					Measured Pre Sample Flow Rate (L/min):	<small>Acceptable Range (0.9 L/min - 1.1 L/min)</small>				
DNPH Cartridge Scheduled Sampling Date/Time:					Leak Check Pass? (Circle One):	<table border="0"> <tr> <td>Yes</td> <td>No</td> <td>Leak Rate (L/min):</td> <td style="text-align: right;"><small>Acceptable Range (&lt;0.03 L/min)</small></td> </tr> </table>	Yes	No	Leak Rate (L/min):	<small>Acceptable Range (&lt;0.03 L/min)</small>
Yes	No	Leak Rate (L/min):	<small>Acceptable Range (&lt;0.03 L/min)</small>							
Field Recovery Information										
Site Operator (initials, date, time of pickup)					Sample Start Date & Time:					
					Sample Stop Date & Time:					
SAMPLE VOLUME (L):					Sample Duration (hrs):					
NIST Flow Standard (S/N and Exp. Date):					Measured Post Sample Flow Rate (L/min):					
Field Comments:										
Laboratory Receipt/Login Information										
Sample Received by:					Sample Receipt Temperature (°C):					
Date and Time Sample Received:					S/N of NIST Traceable IR-Gun:					
Laboratory Comments:										
Extraction Date:				Extracted by (initials):	Extraction Manifold Port Number:					

Appendix C – ATEC 8000 Monitoring Site Logbook Entry Template

North Carolina Department of Environmental Quality, Division of Air Quality Laboratory

ATEC 8000 Monitoring Site Logbook Entry Template			
			<i>Channel #</i>
Date:		Cartridge Sample #:	
Pick up Date:			
Operator ID:		Total Sample Volume	
ATEC 8000 S/N or FAS#:			
BGI-Tetracal S/N and Exp:		Sample Duration (hrs)	
ATEC 8000 Status Flags			
	<i>Channel #</i>		<i>Channel #</i>
Pre-Sampling Flow Rate Reading:		Post-Sampling Flow Rate Reading:	
Site Activities Performed:			
Field Comments:			
Level 1 Review:		Level 2 Review:	

A Printable PDF version of this template is available at **P:\LAB (Laboratory Analysis Branch)\Forms and Templates**