

**STANDARD OPERATING PROCEDURE
FOR
MEASUREMENT OF DIISOCYANATES
USING THE BUCK ELITE-5
SAMPLING PUMP**



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

Acknowledgement

This Standard Operating Procedure (SOP) was developed by EPA Region 4, Science and Ecosystem Support Division. This SOP is based on the Commonwealth of Kentucky's ambient monitoring SOP template. Special thanks to ERG for operational content and illustrations.

For questions or comments please contact:

Greg Noah, EPA, Region 4, SEDS at noah.greg@epa.gov or 706-355-8635

Mike Jones, EPA-OAQPS-AQAD at jones.mike@epa.gov or 919-541-0528

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I. INTRODUCTION

This procedure is designed to provide instruction on collecting ambient diisocyanate compounds using the Buck Elite personal air sampling pump. The procedure follows the tenets/specifications presented in the Occupational Safety and Health Administration's (OSHA) OSHA No. 42.

Samples are collected by drawing a known volume of air through glass fiber filters coated with 0.1 mg of 1-(2-pyridyl) piperazine (1-2PP) which are contained in open-face cassettes. Samples are extracted with 90/10 (v/v) acetonitrile/dimethyl sulfoxide (ACN/DMSO) and analyzed by high performance liquid chromatography (HPLC) using an ultraviolet or fluorescence detector.

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 flow data in its internal memory if data is logged every minute. Four sequential samples over a 24 hour period (*detailed on page 17 of this SOP*) will be collected for this 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 the Eastern Research Group (ERG) directly at 919-468-7800 or by email Julie.Swift@erg.com. Maintenance and troubleshooting should be conducted using the operator's manual.

FIGURE 1. Buck Elite 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 sampling system 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 sampling system on a reasonably level structure with the probe inlet positioned at a height between two (2) and fifteen (15) meters above ground level.

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 sampling pump and collection media (i.e., O₃ scrubber and DNPH Cartridge) must be placed inside an enclosure/storage container to shield it from weather. 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.

ERG has supplied an enclosure to meet the above requirements and is described below. The enclosure and collection components constitute a complete diisocyanates sampling system with the exception of the four sample pump and the system stand. The enclosure is a weather-tight NEMA 4 rated non-metallic unit. Figure 2 presents a view of the outside of the enclosure with four sample collection filter cassettes attached.

FIGURE 2. Diisocyanate 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 main enclosure chassis; attach the enclosure so that the top of the chassis is between 1.5 and 2 meters above ground level. If the enclosure is to be attached to wooden stand material, 4 lag bolts and washers have been supplied for use. If the

stand material is not compatible for attaching with lag bolts, hardware for attaching the enclosure will be the responsibility of the monitoring agency.

2. The sample inlets will now be at the required height specification of at least 2 meters above grade.
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.
4. Everything required to conduct diisocyanates samples collection is included inside the enclosure. Figure 3 presents a view of the inside of the enclosure with one of the four EPA supplied pumps installed.

FIGURE 3. Diisocyanate Sampling System Enclosure



5. Locate the self-adhering Velcro strips inside the enclosure. The loop sides of the strips have been securely adhered to the rear panel of the enclosure. Carefully remove the white plastic material on the hook side of the Velcro strips to expose its associated adhesive. Position a sampling pump in front of each Velcro strip such that the strip runs down the center of the back side of the

pump vertically. Press the pump in against the exposed Velcro strip adhesive to attach it to the back side of the pump. The attached Velcro will help ensure that the pump remains upright during sampling, but will still allow the pump to be easily removed from the enclosure if required.

6. With the enclosure assembled and attached to the stand, level and secure the stand at the site. Sandbags or lag bolts may also be used to secure the sampler.

III. OPERATING PROCEDURE

A. Equipment and Supplies

Buck Elite pump (with back pressure substitute device)
Sampling system enclosure
Tripod or stand
Platinum grade silicone tubing (received from ERG)
Flow meter (calibrated by and received from ERG)
Piperazine preloaded filter cassette
Logbook
Powderfree gloves
ERG sample paperwork
Cooler with ice substitute

B. Initial Receipt Activities

1. Plug sampler into AC power and charge the internal battery for at least 24 hours.
2. Check parts and components against the packing list.
3. After charging, ensure sampler will power up and that the main screen is operational.

C. Installing the Piperazine Preloaded Filter Cassette

The steps presented below should be followed when loading the piperazine preloaded filter cassette shown in Figure 4 in preparation for a sampling episode.

FIGURE 4. 1-(2-pyridyl) Piperazine Preloaded Filter Cassette



1. Remove the protective cap from the collection head.
2. Pull down to completely extend each of the four lengths of platinum grade silicon tubing (silicon tubing) with the syringe tips inserted.
3. Remove the plug from the outlet port of the filter cassette. The outlet plug is blue in color, as shown in Figure 5.

Figure 5. Outlet port of filter cassette



Photograph courtesy of Dave Dayton, ERG

4. Insert the syringe tip into the outlet port of the filter cassette. Ensure that it fits snugly to produce a leak-free connection as shown in Figure 6.

Figure 6. Filter cassette attached to silicon tubing with syringe tip inserted



Photograph courtesy of Dave Dayton, ERG

5. Press the filter cassette upward into the cassette holder, as shown in Figure 7. Repeat steps 4 and 5 for each of the four collection channels. **(Caution: Press only hard enough to ensure that the cassette is securely held. This does not take a large volume of force. Do not press so hard that the cassette cannot be easily removed by simply pulling downward.)**

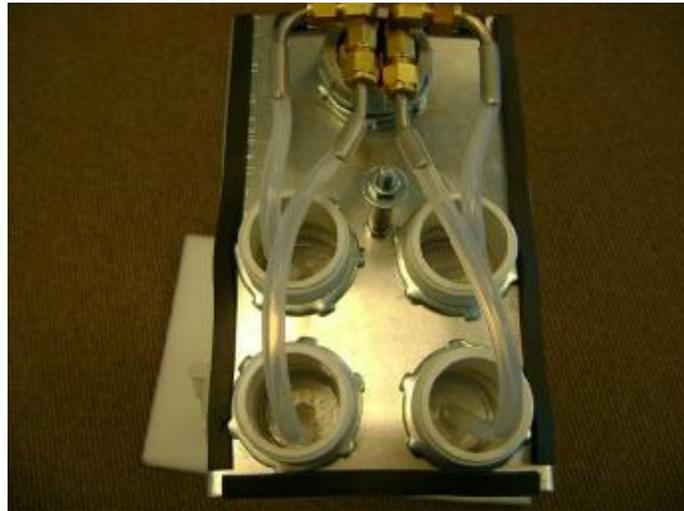
Figure 7. Filter cassette secured in cassette holder



Photograph courtesy of Dave Dayton, ERG

6. Although not a requirement, it would be prudent to be able to look down into each of the cassette holders from above to visually inspect each of the four connections to ensure that they are appropriate, as shown in Figure 8.

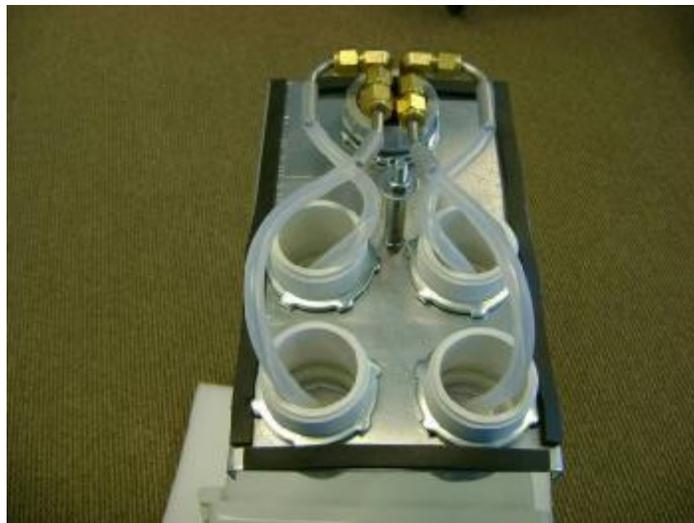
Figure 8. Visual inspection of tubing to cassette outlet port



Photograph courtesy of Dave Dayton, ERG

7. Arrange tubing as shown in Figure 9, to ensure that it will not become crimped when the protective cover is reinstalled.

Figure 9. Tubing arranged to avoid crimping



Photograph courtesy of Dave Dayton, ERG

8. Reinstall protective cover.

9. Remove the plug from the inlet port of the filter cassette. The inlet plug is red in color, as shown in Figure 10.

Figure 10. Inlet port of filter cassette



Photograph courtesy of Dave Dayton, ERG

The system is now ready to measure initial flow rates. To accomplish these measurements, the silicon tubing on the calibrated flow meter is attached to the inlet port of each channel, each pump is actuated, and each measurement is read directly from the scale presented on the flow meter. After collection is completed, post collection flow rates must be measured and the outlet port plug (blue) and inlet port plug (red) on each cassette must be reinserted.

D. Configuring the Buck Elite Pumps

The Buck Elite pumps require a specific minimum volume of back pressure to function properly. The filter cassettes used to collect Diisocyanate samples are zero back pressure media. Accordingly, the pumps must be configured using a back pressure substitute device as supplied by Buck. The back pressure substitute device is a small orifice assembly with a piece of connecting Tygon[®] tubing (see Figure 11-A) that is attached directly to the inlet of each Buck Elite pump (see Figure 11-B). The length of platinum grade silicon tubing that runs to each filter channel is then attached to the inlet of the back pressure substitute device. The back pressure substitute device was provided with each Buck Elite pump supplied by US EPA. It is contained in a plastic bag in the box that the

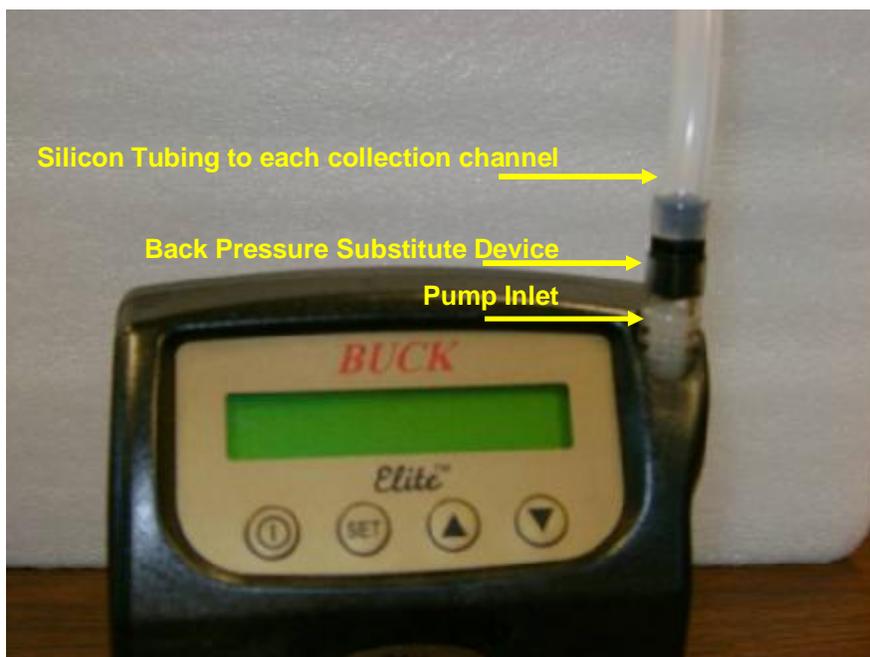
pump was received in and can be identified by the label "DP Adaptor" and part number MK-30229. After attaching the back pressure substitute device to each pump, proceed to setting the flow rate and calibrating the pump per the steps below.

Figure 11-A. Back Pressure Substitute Device (DP Adaptor MK-30229)



Photograph courtesy of Dave Dayton, ERG

Figure 11-B. Installed Back Pressure Substitute Device



Photograph courtesy of Dave Dayton, ERG

E. Setting the Flow Rate

1. Connect the flow meter to the filter cassette by inserting the syringe tip connected to the flow meter into each inlet port.
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 diisocyanate sampling for the study is 900cc/min.

COLLECTION Target = 0.9 LPM (900 cc/min)

4. Releasing the SET key will store the flow rate.
5. Press the ON/OFF key to return to the Main Display.
6. Repeat for the remaining 3 channels.

F. Pump Calibration

To CALIBRATE pump flow:

1. To measure the flow rate connect the flow meter to the inlet port of each channel.
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 collection 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 collection 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.
7. Repeat for the remaining 3 channels.

G. Conducting the Sampling Event

1. Record activities / maintenance in logbook.
2. Prepare sample paperwork. On the RTI Chain of Custody Record complete the top four lines of information and list all four sample ID numbers in the table. Record any pertinent observations on the "Comments" line on the right side of the table.
3. Make sure the flow rate is set and pump calibrated according to the instructions in Section D. Setting the Flow Rate and E. Pump Calibration.
4. Put on a clean pair of powder-free gloves.
5. Inspect the piperazine filter cassette to verify that end caps are in place and have not fallen off through shipping. If end caps are not in place, use a new cassette.

NOTE: If running a field blank, follow steps 3 and 4 above, then remove the end caps from the piperazine filter cassette, count to ten, replace the end caps, and place it back into the antistatic bag. Label the bag to designate the filter cassette as a field blank. Log the filter ID as field blank in the comments section of the RTI Chain of Custody Form.

6. Carefully install new piperazine filter cassettes on all 4 channels according to Section C. Ensure all connections are secure. Install the flow meter at the inlet port of the cassette.
7. Press the down arrow key under the "EXIT" command. The "RUN/MENU" screen should now be displayed.
8. Turn the pump on using the up arrow key below the "RUN" command.
9. Record the flow meter flow rate in the field logbook. Read the center of the ball in the flow meter at "eye level" for the best accuracy.
10. To turn pump off, press and hold the power button until the countdown ceases.
11. Press the down arrow key under the "EXIT" command to return to the "RUN/MENU" screen.

12. Remove flow meter from the inlet probe.
13. 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.
14. Activate Timing Routine - press and hold SET key and press Up arrow twice to get to Start/Stop screen.
15. 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.
16. 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.
17. After Start time is set, press Down arrow to scroll to 5min/hr Cycle where pump can be setup up for 5 min 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.

NOTE: Four sequential samples must be programmed! Each sampler will have a different start and stop time as follows:

Pump 1: 00:01 to 05:01

Pump 2: 06:01 to 11:01

Pump 3: 12:01 to 17:01

Pump 4: 18:01 to 23:01

Sample time programming is critical! ALL SAMPLES MUST SUCCESSFULLY RUN FOR A COMPLETE SAMPLING EVENT.

18. 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.

19. Press RUN key to start sampling. Pump will display Delayed Start Time, then it will shut down within 30 seconds 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 Piperazine Filter Cassettes

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

1. Add flow meter to sample inlet.
2. Press the power button to activate the pump's display.
3. Press the down arrow key to exit the data screen.
4. Press the up arrow key (should be the button under the "RUN" command) to turn on the pump.
5. Record the flow meter flow rate in the field logbook. Read the center of the ball in the flow meter at "eye level" for the best accuracy.
6. Calculate an average flow from the pre and post flow checks. Record the result in the field logbook. Multiply the average flow rate by the sample time to determine a total volume. Record the date sampled, time sampled, and total volume on the RTI Chain of Custody form. Check the "Air" box, provide a sample description, indicate one container, and designate "Diisocyanate" for the analytical parameter on the RTI Chain of Custody form.
7. Remove the filter cassette and immediately cap at each end. Place the capped cassette in a polypropylene bag. The bag must be placed in a cooler cooled to sub-ambient temperature (~4 °C) with ice substitutes and return to RTI for analysis.
8. Repeat for all sampling channels.

Field Data Recovery

1. On the RTI Chain of Custody Form ensure all aforementioned blanks are filled for ALL channels, and the first line of "Relinquished By:" is filled out by the sampler.
2. Data may be downloaded to a laptop using the Buck PC link cable. RTI does not require this data, but direction can be found on pages 15 and 16 in the Buck Elite Instruction Manual document.

NOTE: The data logging feature must be enabled prior to the sampling event.

Sample Shipping

The samples should be in a polypropylene bag along with the paperwork and packed in a small box/cooler. **The samples are required to be shipped cold and at 4 °C.** Use the pre-filled out FedEx label provided by RTI, and fill out the "Sender" section with the sampling agency's address and phone number. Send priority overnight to RTI.

If the shipping form is lost, use the address below for shipping to RTI, and contact them directly for the FedEx accounting.

Address: RTI Laboratories, Inc.
31628 Glendale Street
Livonia, MI 48150-1827
(734) 422-8000

IV. DATA FORMS

All sample related run data forms will be supplied by ERG/RTI. 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 RTI Chain of Custody Form is attached below.

RTI Chain of Custody Form



RTI LABORATORIES, INC.

CHAIN OF CUSTODY RECORD



PAGE:	CF:
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MAIN LAB & HEADQUARTERS
RTI LABORATORIES, INC.
31628 Glendale Street
Livonia, MI 48150-1827
Phone (734) 422-8000
Fax (734) 422-5342
www.rtilab.com

Please Include Email Address of Report Recipient Whenever Possible !!!

SUBMITTING COMPANY:				REPORT TO:				BILL TO:											
PROJECT NAME:		PROJECT #:		QUOTE #:		COMPANY:													
SPECIAL INSTRUCTIONS / COMMENTS:						PHONE:													
						FAX:				EMAIL:									
SAMPLER'S PRINTED NAME:				SAMPLER'S SIGNATURE:				ANALYTICAL PARAMETERS				COMMENTS Methanol Preserved Weights HOT Sample Notation etc.							
ITEM #	SAMPLE I.D.	DATE SAMPLED	TIME SAMPLED	AIR	SOLID	FLUID	VOLUME	SAMPLE DESCRIPTION	NUM OF CONTAINERS										
1																			
2																			
3																			
4																			
5																			
6																			
7																			
8																			
9																			
10																			
Relinquished By:		Date	Time	Received By:		Date	Time					FOR LAB USE ONLY Were samples preserved <input type="checkbox"/> in field <input type="checkbox"/> in lab <input type="checkbox"/> N/A Were samples filtered <input type="checkbox"/> in field <input type="checkbox"/> in lab <input type="checkbox"/> N/A Temp of samples _____ °C On Wet Ice ? _____ Comments: _____							
Relinquished By:		Date	Time	Received By:		Date	Time												
Relinquished By:		Date	Time	Received By:		Date	Time												
TAT: Standard <input type="checkbox"/>				RUSH: Next BD <input type="checkbox"/> 2nd BD <input type="checkbox"/> 3rd BD <input type="checkbox"/>															
				Note: RUSH requests will incur surcharges!															
Distribution: White and Yellow - Lab; Pink - Field								See reverse side for Laboratory Terms and Conditions of service											