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The Arizona Border Study

An Extension of the
Arizona National Human Exposure Assessment Survey (NHEXAS)Study
Sponsored by the Environmental Health Workgroup of the Border XXI Program

Quality Systems and Implementation Plan for Human Exposure Assessment

The University of Arizona
Tucson, Arizona 85721

Cooperative Agreement CR 824719

Standard Operating Procedure **SOP-UA-F-11.1**

Title: Use of an Active Sampling Device for the Collection of Airborne VOCs at Fixed Indoor and Outdoor Sites

Source: The University of Arizona

U.S. Environmental Protection Agency
Office of Research and Development
Human Exposure & Atmospheric Sciences Division
Exposure & Dose Research Branch

Title: **USE OF AN ACTIVE SAMPLER FOR THE COLLECTION OF AIRBORNE VOCs AT FIXED INDOOR AND OUTDOOR SITES**

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Form TP-1

Use of an Active Sampling Device for the Collection of Airborne VOCs at Fixed Indoor and Outdoor Sites

1.0 PURPOSE AND APPLICABILITY

This standard operating procedure (SOP) describes the methods used to collect indoor and outdoor air samples for the determination of selected volatile organic compounds (VOCs) using a pump to draw air through a cylindrical tube (Carbotrap) packed with a carbon-based mixed sorbent. Volatile organic compounds (VOCs) present in the air are preferentially adsorbed during the sampling period and remain so until thermally desorbed into a gas chromatograph/mass spectrometer system for characterization (see SOP BCO-L-22.X for details of analysis). This procedure is used in support of the NHEXAS Arizona Project, AZ Border Project (Border AZ) and other Health and Environment projects.

2.0 DEFINITIONS

2.1 ACTIVE SAMPLER = collects contaminant by actively drawing air through the sorbent contained in the sampler using a pump.

2.2 AZ Border = The US border region is defined as 100 km north of the border. In this study, we define the border as 40 km north of the border. The Arizona Border Study or "Border AZ" is an alias for "Total Human Exposure in Arizona: A Comparison of the Border Communities and the State" conducted in Arizona by the University of Arizona / Battelle / Illinois Institute of Technology Consortium.

2.3 BUCKET = A plastic container with a buckle top or tight-fitting lid. One bucket is assigned to each household to be sampled. Household identification and stage numbers are listed on the outside of the container. The bucket contains all paperwork and questionnaires to be completed by field staff or household respondents. It serves as the primary vehicle for securing and transporting forms, data and samples to and from the field through the course of the study.

2.4 CARBOTRAP = A cylindrical tube packed with a carbon-based mixed sorbent which preferentially collects VOCs from the sample environment.

2.5 CHAIN OF CUSTODY RECORD (Fig.1) = A vital data tracking and quality assurance form which accompanies every sample.

2.6 DATA COORDINATOR = The employee of the research project who supervises data batching, entry and verification.

2.7 FIELD COORDINATOR = The employee of the research project who supervises field data collection and operations. The Field Coordinator collates HH specific data into HH packets, and upon completion of all visits, sampling and QA checks, forwards the packet to the Data Coordinator.

2.8 FIELD KIT = A sampling tool-box containing appropriate collection and storage utensils. For the active collection of airborne VOCs at fixed indoor and outdoor sites the kit should include: SKC pumps, low flow controllers, diffusion limiting caps, non-sterile and non-powdered latex gloves, bubble flow meters, a psychrometer with three charged D cell batteries, crescent and channel-lock wrenches, and extra copies of the Active VOC Sampling Data Sheet (Fig. 2).

2.9 FIELD STAFF = The Field Coordinator, the Team Leader and the Team Members.

2.10 HRP OFFICE = The **H**ealth **R**elated **P**rofessions building, currently located at 1435 North Fremont Avenue, Tucson, AZ 85719. This is an annex of the Arizona Prevention Center and the primary site of the operations for NHEXAS Arizona project, AZ border project and other Health and Environment projects.

2.11 HOUSE**H**OLD(HH) = The residence occupied by study respondent(s).

2.12 HOUSE**H**OLD **ID**ENTIFICATION NUMBER(HHID) = A unique number and character combination which is assigned to each respondent household for identification purposes. This number must be recorded on all data (forms, samples, questionnaires and correspondence) related to the household.

2.13 LAB SUPERVISOR = The employee of the research project who supervises laboratory analyses.

2.14 MATERIALS TECHNICIAN (Materials Tech) = The employee of the research project who is responsible for assembling and assigning field forms, questionnaires and equipment for field use. The Materials Tech assigns each sorbent tube a unique sample ID number upon receipt from Battelle.

2.15 N/A = Not Applicable.

2.16 NHEXAS Arizona = Acronym for **N**ational **H**uman **EX**posure **A**ssessment **S**urvey, a research project conducted in Arizona by the University of Arizona/Battelle/Illinois Institute of Technology consortium.

2.17 PACKET = A sturdy, envelope-like container that can be fully closed and is large enough to hold the physical data forms generated from sampling and surveying a study household.

2.18 QUALITY ASSURANCE (QA) = All those planned and systematic actions necessary for ensuring the accuracy, validity, integrity, preservation and utility of collected data.

2.19 QUALITY CONTROL (QC) = Those quality assurance actions providing a means to control and measure the characteristics of a datum, process or the adherence to established parameters.

2.20 RESPONDENT = A person in the study population of NHEXAS Arizona project, AZ border project or other Health and Environment projects. Each household is assigned a HHID number. All of the respondents are assigned an Individual Respondent Number (IRN). Each respondent can be uniquely identified by a HHID, and IRN combination.

2.21 SAMPLE = The VOCs absorbed by the Carbotrap during sampling.

2.22 SAMPLE IDENTIFICATION NUMBER = A numeric code that uniquely identifies every sample. It is generated by the NHEXAS tracking system by the Materials Technician at the HRP Office when the material is logged-in to the Tracking System.

2.23 SKC PUMP = Is an Aircheck personal air sampling pump produced by SKC. The pump comes with a 115 V charger. The pump is programmed by the Field Team to draw ambient air through the Carbotrap at 5 mL/min.

2.24 SOLID SORBENT = Material used to collect selected vapor phase organic compounds (VOCs) by drawing ambient air through a cartridge containing the material. Analysis is typically by thermal desorption of the components into a gas chromatograph/mass spectrometer (GC/MS) system.

2.25 TEAM LEADER = The member of the field team who is primarily responsible for respondent contact, data collection, field form and questionnaire completion, and site QC checks of all data.

2.26 TEAM MEMBER = Member of a field team responsible for assisting the team leader in the collection of data and quality control checks in the field.

2.27 TRACKING SYSTEM = A database system containing information about the custody, transfer and storage of hard copy data, electronic data, field samples, and field sample aliquot.

2.28 VISIT = A scheduled appointment with participating respondents at their place of residence (HH) for the collection of samples, questionnaires and other data.

3.0 REFERENCES

3.1 A.J. Pollack, S.M. Gordon, and D.J. Moschandreas. March 1993. *Evaluation of Portable Multisorbent Air Samplers for Use with an Automated Multitube Analyzer.* **Report EPA/600/R-93/053,** U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, 91 pp.

3.2 P. Ciccioli, A. Cecinato, E. Brancaleoni, M. Frattoni, and A. Liberti, *Use of Carbon Adsorption Traps Combined with High Resolution Gas Chromatography-Mass Spectrometry for the Analysis of Polar and Non-Polar C_4-C_{14} Hydrocarbons Involved in Photochemical Smog Formation.* **J. High Res. Chromatogr.,** 15, 75-84 (1992).

3.3 R.W. Bishop and R.J. Valis, *A Laboratory Evaluation of Sorbent Tubes for Use with a Thermal Desorption Gas Chromatography-Mass Selective Detection Technique,* **J. Chromatogr. Sci.,** 2, 589-593 (1990).

3.4 Lebowitz, M.D. 1993. *Study Design* (Revision of 31 Dec. 93). **EPA NHEXAS Cooperative Agreement.**

4.0 DISCUSSION

4.1 VOCs in the air can be adsorbed, retained, concentrated, and removed from solid sorbents in order to facilitate their identification and quantification. In practice, this is accomplished by drawing the air to be sampled through a tube packed with a suitable solid sorbent. Gaseous organic species are preferentially adsorbed during the sample collection phase, which is designed to pass a fixed volume of air through the cartridge at a known flow rate using a pump. After the samples are returned to the laboratory, they are thermally desorbed into a combined GC/MS system for analysis. For the present study, the VOCs of interest are benzene, toluene, trichloroethylene, and 1,3 butadiene.

4.2 The relative timing of actively pumped VOC samples to other sample types collection is shown in figure 3.

5.0 RESPONSIBILITIES

5.1 Battelle personnel are responsible for:

(a) purchasing carbotraps,
(b) cleanup of unexposed carbotraps before shipment for field use,
(c) shipping carbotraps in pre-packaged units to the HRP Office for use in household sampling,
(d) assuring that blank levels for each batch of carbotraps meet acceptability requirements prior to shipping to the HRP Office,

(e) documenting blank levels in a NHEXAS (or other study) laboratory notebook.

5.2 The Materials Technician is responsible for:

(a) receipt of carbotrap samples shipped by Battelle personnel,
(b) logging the material into the Tracking System and generating a unique sample ID for each sampler in accordance with SOP UA-G-5.X,
(c) storing the carbotraps in the pre-field storage bin at four degrees centigrade (4°C) before assignment to the HH,
(d) assigning blanks and samplers to the field and documenting the assignment in the Tracking System,
(e) shipping the exposed samplers and blanks on blue-ice with the appropriate custody documentation to Battelle for analysis.

5.3 The Field Coordinator is responsible for;

(a) supervision of Field Staff
(b) performing a 10% QA audit of actively pumped voc sampling in the field.
(c) 100% QA check of all Active VOC Sampling Sheets within 24 hours of submission by the Team Leader

5.4 The Team Leader is responsible for:

(a) sample site selection,
(b) custody of all samples until they are transferred to the Field Coordinator.

5.5 The Team Member is responsible for:

(a) Sample collection according to the procedures outlined in this SOP,
(b) Documenting the collection on the field sheet (Fig. 2) and the Chain of Custody Record (Fig. 1).

6.0 MATERIALS AND EQUIPMENT

6.1 Materials

(a) Carbotrap 300 Thermal Desorption Tubes (Supelco,Catalog No. 20379), containing 20/40 Carbotrap C (300 mg), 20/40 Carbotrap B (200 mg), and 60/80 Carbosieve S-III (125 mg), in 11.5 cm x 4 mm id x 6 mm OD glass.
(b) Airchek (SKC) air sampling pump (Model 224-52) with 115 V charger and (single or dual) low flow constant pressure controller for low flow applications (5-500 mL/min); or Air Cadet (Barnant) vacuum-pressure pump with low flow constant pressure controller.

(c) Shipping containers / biomailers.
(d) Non-sterile, non-powdered latex gloves.
(e) Stopwatch.
(f) Tweezers.
(g) Glass fiber filters (Gelman Sciences, or equiv).
(h) Bubble flow meter, 10 mL, Mini-Buck Digital Bubble Flow Meter, or Sierra Digital Mass flow meter.
(i) Tape and rain hood for outdoor sampling.
(j) Clean glass wool packing material.
(k) Kim wipes.
(l) Logbook and data sheets.
(m) Psychrometer and 3 'D' cell batteries.
(n) Cooler and blue-ice.
(o) Soap solution.

7.0 PROCEDURE

7.1 Preparation

7.1.1 Field Site Selection Criteria

A. INDOOR SITE SELECTION

(a) Sampling sites are chosen by the Team Leader. Indoor sampling occurs in the same room that PM (UA-F-3.X) and Passive VOC sampling (UA-F-12..X, UA-F-13.X) is conducted.
(b) Situate the sampling equipment in a main living area of the home. The main room is the room where the HH members spend the majority of their time when indoors. Bedrooms and private areas where the pump may disturb participants are to be avoided.
(c) Place the sampler as close as possible to the center of the room, but minimize the inconvenience to the respondents. The sampling inlet line should be placed approximately 4 - 6 feet above the floor, approximately 2 feet from any corners or walls.
(d) Avoid placement near windows, air conditioners, and other ventilation devices. Avoid stagnant zones or direct drafts. The sampler must be placed as far as possible from obvious sources of contamination such as naked pilot lights or gas heaters.
(e) Try to avoid locating the sampler close to the passive sampling set-up (UA-F-12.X, UA-F-13.X) and fixed site PM stands (UA-F-3.X). However, space to set-up equipment and access to power outlets will be limited and compromises will need to be made. Record the sampler location on the field sheet.
(f) Once a suitable site is chosen confer with the HH respondents and insure that the

selected location is acceptable to the participants. Explain your location decision as necessary and find a mutually agreeable site.

B. OUTDOOR SITE SELECTION

(a) The Active VOC set-up should be placed outdoors on the North side of the HH, at least ten feet from the midpoint of the wall and four to six feet above the ground. Placement on the north side of the home is intended to protect the sampler from direct sunlight.
(b) If the North side of the HH faces a street or places the sampler at risk for theft or vandalism, place the sampler in a more secure part of the HH property. Indicate the location on the field data sheet (Fig. 2)
(c) The Active VOC pump makes a noise equivalent to a fish-tank pump and should be kept away from sleeping quarters where possible.
(d) Do not locate the sampler under trees, near pools of standing water, near animal cages or under tables, etc.
(e) Do not locate the sampler near obvious sources of contamination such as roads, alleys, barbecue pits, etc.
(f) The Active VOC sampler may need to be located on a the same sampling stand as the passive VOC (UA-F-12.X, UA-F-13.X) and fixed site PM stands (UA-F-3.X). due to limited workspace and access to power outlets. Record the sampler location on the field sheet.

7.1.2 Notes of Caution

(a) The Solid Sorbent is very easily contaminated. Use great care in handling and transporting the carbotraps at all times. Carbotraps must be kept at $4^{O}C$ or on blue-ice except during sample collection. Do not store carbotraps at $-20^{O}C$ as extreme freezing and thawing may serve as a pump to expel collected VOCs and invalidate samples.
(b) Be sure that swagelok fittings are snugly applied at all times. Caps and connectors should be made hand-tight and then advanced an additional one quarter turn using a set of crescent wrenches. The sorbent will begin adsorption immediately upon un-capping the carbotrap - even when no air is being actively drawn across the sorbent bed by the SKC pump.
(c) Do not write or affix anything to the sampler tube itself.
(d) Never touch a carbotrap with bare hands as contamination may result. Handle carbotraps with non-sterile, non-powdered latex gloves, tweezers or wrenches only.
(e) Never use any tube dropped or scraped against any surface. If a tube is suspected of being contaminated, mark the carbotrap appropriately and use an alternate.
(f) Sampling should not be started during rainstorms as samples taken during active rain periods contain too much moisture to be analyzed on take-down, the Team

Leader will inquire as to the local weather conditions during the sampling period.

(g) Do not crimp or cut the diffusion limiting tubing. The length of the tube is determined by Battelle and should not be altered. Battelle will supply replacements as necessary.

7.1.3 Reagents - N/A

7.1.4 Standards and Blanks

Ten percent of all samples collected will be for QA and QC purposes. Field Blanks, Lab Blanks and Spike Blanks will undergo the same preparation, transportation, site set-up, collection and post-field storage and handling as the accompanying active samplers. Dual adjustable low flow controllers will be used for duplicate or replicate sampling.

7.1.5 Pre-Field Checks

A. SKC PUMPS

(a) SKC pumps have a battery which can supply sufficient power for 8 hours of active pumping (pump period) over a 72 hour run time (sample period). The pumps can also be connected to a wall outlet and run for extended periods of time. Indeed, the pump will function as long as it is connected to a reliable source of power. Preliminary testing at HRP has shown that SKC pumps can pump for 9999 minutes out of a 9999 sample period when connected to an AC adaptor. This upper limit is due to the fact that the LCD display is limited to four digits.

(b) Active VOC Sampling is conducted with SKC Pumps that are connected directly to AC power sources inside and outside the sampling location. However, active VOC samples can also be collected in situations where AC power is not available.

(c) If sampling is to be conducted at a location where AC power is not available:

1. The SKC pump batteries must be completely drained before recharging. Recharging times can vary from 12 to 16 hours. The pump will maintain a full charge for 5 - 7 days after charging.
2. The green-light on the unit recharger will light when charging is complete.
3. Battery charging is recorded on the SKC Pump Log (Figure 4).
4. The SKC Pumps are placed in the Household Bucket and are ready for use in the field.

(d) If sampling is to be conducted at a location where AC power is available, two SKC pumps with AC adapters are assigned to the HH by the Materials Technician and placed in the household bucket before sample collection.

(e) Pumps are periodically flow checked and submitted to the Field Coordinator for routine maintenance. Flow rates for active VOC sampling are controlled by the low flow controller and not the SKC pump per se, thus periodic checks of SKC pumps are acceptable. Pump checks (when completed) are documented on the

Low Flow Controller and Pump Check Log (Figure 5).

B. LOW FLOW CONTROLLERS

(f) Low flow controllers are initially calibrated using the following procedure. Periodic calibration may be necessary.

(g) The sampling train is assembled at the UA Field Staging Area. The train consists of an SKC pump, a low flow controller, a sample carbotrap, a diffusion limiting cap and a flow meter. Periodic low flow controller checks are documented on the Low Flow Controller Check Log (Figure 5).

(h) Turn the SKC pump on using the ON/OFF switch, the START/HOLD key and the FLOW AND BATTERY CHECK key. Adjust the flow to 2 Lpm using a Sierra digital mass flow meter or the Mini-Buck digital bubble flow meter.

(i) Press the FLOW AND BATTERY CHECK key to set the pump on hold.

(j) Connect the pump to the bubble flow meter if the 2 Lpm check was performed with the Sierra digital mass flow meter. If the 2 Lpm check was performed using the Mini-Buck, leave the pump connected to it.

(k) Connect the Carbotrap cartridge that has been set aside for flow checks to the low flow controller and cap the distal end with the diffusion limiting tubing. Be sure that all connections are snug.

(l) Connect the low flow controller to the bubble flow meter or the Mini-Buck.

(m) Press the START/HOLD key to start the pump and let run for a minute. Measure the sampler flow rate using the bubble flow meter and stop watch, or digital flow meter.

(n) Calculate flow rate by using the following formula:

FLOW RATE (mL/min) = [Bubble flow meter volume (ml)/elapsed time (s)] x [60 (s)/1(min)]

(o) Adjust the flow if the measured flow rate is not within 8-12 mL/min. Flow is set on the adjustable low flow holder by turning the screw (needle valve) in the top of the holder. Clockwise rotation of the screw closes the needle valve and decreases flow rate and counter clockwise rotation opens the valve and increases flow rate.

(p) If the flow is adjusted, allow the pump to run for one minute at the new setting and repeat steps (m) and (n).

(q) Repeat (m) - (p) until three consecutive readings within 8-12 mL/min are recorded.

(r) If a dual adjustable low flow controller unit is to be used for duplicate sampling attach the sample carbotrap to one line and repeat steps (l) through (q) above. Then attach the carbotrap to the second line and repeat steps (l) through (q) above.

(s) Due to the individual nature and variable flow characteristics of each carbotrap, pre-field calibration of the low-flow controllers will not guarantee that the same flow rate will be later be achieved using a different carbotrap in a different environmental setting. Pre-field calibration of pumps and carbotraps helps keep the carbotraps in 'the right ball-park', and thus is conducted periodically.

7.2 Field Procedures

TROUBLESHOOTING:

Flow faults encountered on set-up are most likely the result of a crimp or fold in the black rubber connector linking the tygon sample tubing and the SKC pump inlet jet. Clear the fault by adjusting the tubing and restart. For additional troubleshooting hints see Figure 6., *The Active VOC Sampling Troubleshooting Guide.*

7.2.1 Standards and Blanks Deployed

(a) The Field Blank for active VOC sampling will undergo similar preparation, transportation, site setup, collection and post-field storage conditions as the accompanying active sampler, but the blank will not be connected to the SKC pump or low flow controller.
(b) At the sampling site, one end of the blank carbotrap will be uncapped and immediately recapped. The ample-ID of the blank will then be recorded on the field sheet.
(c) The Field Blank carbotraps will otherwise be treated the same as a 'live' sample. They will be transported in a cooler to the HH under appropriate conditions.
(d) Duplicate sampling will be accomplished by using a duplicate low-flow controller which draws air through both tubes at the same time.

7.2.2 Samples

A. SETUP AND FLOW CHECKS

(a) Verify sample site suitability with the Team Leader and note any potential contaminant sources within 10 feet of the site. Remove the anti-tamper plate.
(b) Turn the SKC pump on using the <u>ON/OFF</u> switch, the <u>START/HOLD</u> key and the <u>FLOW AND BATTERY CHECK</u> key. Adjust the flow to 2 Lpm using a Sierra digital mass flow meter or the Mini-Buck digital bubble flow meter.
(c) Press the <u>FLOW AND BATTERY CHECK</u> key to set the pump on hold.
(d) Connect the pump to the bubble flow meter if the 2 Lpm check was performed with the Sierra digital mass flow meter. If the 2 Lpm check was performed using the Mini-Buck, leave the pump connected to it.
(e) Open the cooler and remove a carbotrap. Label the Ziploc bag housing the carbotrap with the HHID, Sample-ID, Start Date, and Tech ID.
(f) Put-on a pair of disposable latex gloves and remove the carbotrap from its container/packaging. Verify that the **HX** serial number engraved on the carbotrap is recorded with the sample-ID on the field sheet.

(g) Remove one end cap from the carbotrap and connect it immediately to either the diffusion limiting cap or low flow controller swagelok fittings. Be sure that the connection is hand-tight plus one half to three quarters of a full turn using a pair of crescent wrenches.

(h) Be sure that the arrow on the carbotrap is pointing toward the low-flow controller and the pump unit. Connect the second fitting and verify that all connections are snug. The order of hook-up does not matter, as long as the arrow on the carbotrap is pointing toward the low flow controller and the SKC pump.

(i) Place all carbotrap fittings and connectors in the carbotrap housing and store them in the HH bucket until the samples are picked up in 3 days time.

(j) Connect the low flow controller to the bubble flow meter or the Mini-Buck.

(k) Press the START/HOLD key to start the pump and let run for a minute. Measure the sampler flow rate using the bubble flow meter and stop watch, or digital flow meter.

(l) Calculate flow rate by using the following formula:

FLOW RATE (mL/min) = [Bubble flow meter volume (ml)/elapsed time (s)] x [60 (s)/1(min)]

(m) Adjust the flow if the measured flow rate is not within 8-12 mL/min. Flow is set on the adjustable low flow holder by turning the screw (needle valve) in the top of the holder. Clockwise rotation of the screw closes the needle valve and decreases flow rate and counter clockwise rotation opens the valve and increases flow rate.

(n) If the flow is adjusted, allow the pump to run for one minute at the new setting and repeat steps (k) and (l).

(o) Repeat (k) - (n) until three consecutive readings within 8-12 mL/min are recorded. The flow rate recorded on the field sheet is:
   1. The average of 3 consecutive bubbles between 8 and 12 Ml/min when flow checks are made with a hand-held burette or the MiniBuck Digital Bubble Flow Meter, or
   2. The reading on the digital display one minute after the flow rate stabilizes between 8 - 12 mL/min when using the electronic mass flow meter.

(p) If a dual adjustable low flow controller unit is to be used for duplicate sampling attach two carbotrap tubes and proceed with steps (k) - (o) for both tubes.

(q) Once the flow-rate has been adjusted (as necessary) and recorded, pause the SKC pump unit by pressing 'HOLD'.

(r) Record the temperature, relative humidity and calibration time on the field data sheet (Fig. 2).

B. SKC PUMP PROGRAMMING

When programming the SKC pump, use the DELAYED START function to pause the sampler for as long as necessary. The diffusion limiting cap will limit carbotrap contamination while the pump is not active. The pump should be programmed so that it will remain paused until 24 hours before the return visit of the field team (Fig. 3). The delayed start would be 48 hrs in a typical stage 3 scenario followed by a 24 hr. run time.

(a) While the pump unit is paused (the HOLD indicator should be flashing on the LCD display) press the SET-UP key.
(b) DELAYED START will flash on the display. Use the SET DIGIT key to increment the flashing character and the SELECT DIGIT to switch from one order of magnitude to the next.
(c) Pre-field trials at Battelle have shown that the optimal sampling cycle for the NHEXAS project is a 48 hr. delayed start followed by a 24 hour sample period during which the pump runs for 1 minute, the pauses for two minutes. The set-up is then collected by the field teams within 24 hours of sample collection. (see Figure 3).
(d) Program the delayed start for 2880 minutes (60mins. x 48 hrs), unless otherwise instructed by the Team Leader.
(e) Press the MODE key and the SAMPLE PERIOD prompt will be flashing. Use SET DIGIT and SELECT DIGIT to program a sample period of 1440 minutes (60 mins. x 24 hrs).
(f) Press the MODE key and program the PUMP PERIOD. The pump period is the actual number of minutes you want the pump to actively sample before automatic shutdown. Program the PUMP PERIOD for 480 minutes (60 mins. x 8 hrs). The SKC pump will automatically calculate and control the integrated run time. That is, the pump will program itself to start in 48 hours and once started, it will sample for one minute and pause for two over a 24 hour period.
(g) Verify that the pump settings are correct by scrolling through the values using the MODE key.
(h) Start the sample cycle by depressing the START/HOLD key and DELAYED START will flash on the LCD display.
(i) Re-attach the anti-tamper plate and verify that there are no knots or kinks in the sample lines.
(j) Protect outdoor SKC set-ups by shielding the SKC pump with a rain-hood or enclose the pump unit in a metal toolbox.

C. SAMPLER AND SAMPLE RETRIEVAL

(a) The active VOC sampler will shut itself off automatically at the end of the sampling period.
(b) Locate the sampler and record any changes in the sample set-up or configuration that you notice on the field data sheet.
(c) Record flow fault or other error messages as appropriate.
(d) Remove the anti-tamper plate and record the pump run time by depressing the PUMP RUN TIME key on the sampler. The pump run time is the total active

sampling or exposure time for the carbotrap and should be 480 mins.

(e) The total elapsed time (including the delayed start) is found by depressing the TOTAL ELAPSED TIME key. The total elapsed time should be close to 4320 mins.

(f) Record the PUMP RUN TIME and the TOTAL ELAPSED TIME in addition to the take-down temperature and relative humidity on the field data sheet (Fig. 2).

(g) Start the pump and allow it to run for 2 minutes before obtaining a take-down flow rate.

(h) After two minutes, the flow rate to be recorded on the field sheet will be:
   1. The average of 3 consecutive when flow checks are made with a hand-held burette or the MiniBuck Digital Bubble Flow Meter, or
   2. The reading on the digital display three minutes after the pump was turned on when using the electronic mass flow meter.

(i) Ask the respondents for an approximation of the total number of hours of local rainy weather which overlapped with sample collection and record their answer on the field data sheet.

(j) The Team Leader will record the number of hours of rainy weather which overlapped with sample collection on the field data sheet. Battelle personnel will make the determination as to whether the number of rainy hours is sufficient to void the sample.

(k) Disconnect the sampling train one end at a time. Move as quickly and as smoothly as possible. Insure that swagelok caps are tightened. Place carbotrap in original container and place in cooler.

(l) Return to the University of Arizona on blue ice and store at 4C. The carbotraps must be shipped to Battelle for analysis within four weeks of collection. Shipments will be made to Battelle weekly.

7.3 Calculations

7.3.1 The calculation to determine flow rate in milliliters (mL) per minute is as follows:

$$\text{Flow Rate (mL/min)} = \frac{\text{Flow meter volume(mL)}}{\text{Elapsed Time(s)}} \; \frac{* \; 60\text{(s)}}{1 \text{ min}}$$

7.3.2 The calculation to determine the arithmetic average flow rate is as follows:

$$\text{Average Flow (mL/min)} = \frac{\text{Sum of 'N' Flow Rates (mL/min)}}{\text{'N'}}$$

7.4 Quality Control

Field teams consist of 2 - 3 Team members assigned to different tasks when in the HH. On the Active VOC Sampling Data Sheet (Figure 2), there are double check points at many critical data entry/recording moments. These opportunities serve as an independent verification of the data and the readings recorded. The Team Member independently verifies the values recorded by their team-mate and records a "✓" in the appropriate box.

Once the Field Team Member has completed the set-up in either the indoor or outdoor environment, she or he switches with a second Field Team Member and verifies the readings recorded for the alternate location. Ten percent of all the samples collected will be for QA and QC purposes.

7.4.1 Tolerance Limits

(a) The measured flow rate through the low flow controller should be approximately 10 mL/min. A reading between 8mL/min and 12mL/min on three consecutive occasions is acceptable for the set-up. Record any flow during take-down.

(b) The total elapsed time should be 4320 minutes unless the pump experiences a flow fault or other problem. The pump run-time should be 480 minutes unless the pump experiences a flow-fault or other problem.

(c) Sampler site location is flexible within the bounds of safety and security. Whenever site criteria are not met the field team must annotate the field sheet appropriately.

7.4.2 Detection Limits

(a) The bubble flow meter will be read to the smallest minor division of the meter.

(b) "Seconds" used in flow calculations will be read to within 0.1 seconds. "Minutes" on the SKC pumps are recorded as whole minutes. Decimal, fractions or portions of a minute are not recorded by the pump and are therefore undetectable.

(c) The number of rainy hours will be an approximation based on the anecdotal testimony of the household respondents. The convectional rain systems that Arizona experiences indicate that the information obtained from the respondents as to the local weather in the past several days will be more accurate than using regional chartological data.

7.4.3 Corrective Actions

a) Low flow controllers are adjusted between 8 mL/min and 12 mL/min. Controllers which cannot be calibrated to within this range must be red-flagged and handled in accordance with UA-G-2.X.

b) Apparent mislabeling problems detected in the field may be corrected by the Team Members when appropriate and in accordance with SOP #UA-C-2.X.

8.0 RECORDS

8.1 Chain of Custody Record (Figure 1).This Record will serve as the primary record of sample custody. The Team Leader and the Field Team are responsible for the thorough completion of this form. The completed original Chain of Custody Record will remain with the data sample except when the filters are left at a HH while sampling is taking place. The Chain of Custody Record will be stored with the appropriate field sampling sheet in the HH Bucket until the filter is collected from the field. The custody record will then be reunited with the sample by the Team Leader.

8.2 Active VOC Sampling Field Data Sheet (Figure 2)

8.2.1 This sampling data sheet serves as a record of critical field operation and tracking information for Active VOC sampling. The data sheet (Fig. 2) serves as the primary record on in-field observations and activities.

8.3 Relative timing of Active VOC sampling (Figure 3). This diagram documents the relative timing of active VOC sampling to other sample collection activities for NHEXAS AZ.

8.4 SKC Pump Log (Figure 4)

8.4.1 This data form documents pump charging and discharge cycles. Hours of operation and problems encountered are documented on this form. The forms are stored with their respective SKC pump document file.

8.5 Pre-Field Low Flow Controller Check Log (Figure 5)

8.5.1 This form is completed upon initial receipt of low flow controllers. Periodically, controllers may need to be re-calibrated and a new form will be completed to accomplish this.

8.6 Active VOC Sampling -Trouble shooting Guide (Figure 6)

8.7 Figure 7.: Battelle VOC sampling data. These data were used to determine which sampling strategy would best meet the needs of the NHEXAS AZ study design by minimizing the burden on subjects and maximize data return from field. Based on these data it was decided that SKC pumps would be paused for 48 hours, sample for 24 hours, and be collected within 24 hours without sample loss or contamination.

Figure 1. Chain of Custody Record

Chain of Custody Record
NHEXAS Arizona Project (CR-821560)
Respiratory Sciences
1435 N. Fremont Ave
Tucson, AZ 85719
(520) 626 - 4226

Sample Type: ______________________. page ____ of ____.

Generated by: ______________________/______________________
print name signature

Date Generated	Time	Sample ID	# of Containers	Remarks
__/__/__	__:__			

History of Sample Handling and Custody

Relinquished or Received	Signature	Date mo / day / yr	Time	Action
[Rel] or [Rec]		__/__/__	__:__	
[Rel] or [Rec]		__/__/__	__:__	
[Rel] or [Rec]		__/__/__	__:__	
[Rel] or [Rec]		__/__/__	__:__	
[Rel] or [Rec]		__/__/__	__:__	
[Rel] or [Rec]		__/__/__	__:__	
[Rel] or [Rec]		__/__/__	__:__	
[Rel] or [Rec]		__/__/__	__:__	
[Rel] or [Rec]		__/__/__	__:__	
[Rel] or [Rec]		__/__/__	__:__	
[Rel] or [Rec]		__/__/__	__:__	
[Rel] or [Rec]		__/__/__	__:__	
[Rel] or [Rec]		__/__/__	__:__	
[Rel] or [Rec]		__/__/__	__:__	
[Rel] or [Rec]		__/__/__	__:__	
[Rel] or [Rec]		__/__/__	__:__	

FORM = UA-G4 0 - 1 2

Attach Additional Pages as Necessary

Figure 2. Active VOC Sampling Data Sheet

Active VOC Sampling Data Sheet

Study	Stage #	Team	HHID	F.S.	Visit
● 1 NHEXAS ○ 2 Border ○ 3 ____ ○ 4 ____ ○ 5 ____	3 Collapsed? Y ○ N ● 8 ○	Leader: ____ Init. Tech ID QC✓ By: ____ Init. Tech ID	Sampling Date MO / DAY / YR		QC: ✓ [ ]

[ ] Indoor or [ ] Outdoor

	Setup	Teardown	Setup QC	Takedown QC
Date			☐ ✓✓	☐ ✓✓
Time:			☐ ✓✓	☐ ✓✓
Pump ID			☐ ✓✓	☐ ✓✓
Controller ID			☐ ✓✓	☐ ✓✓
Sample ID	27 -	27 -	☐ ✓✓	☐ ✓✓
Sample Series	HX-	HX -	☐ ✓✓	☐ ✓✓
Temperature	°C or °F	°C or °F	☐ ✓✓	☐ ✓✓
RH	%	%	☐ ✓✓	☐ ✓✓
Flowmeter ID			☐ ✓✓	☐ ✓✓
Flow	mL/min	mL/min	☐ ✓✓	☐ ✓✓
Cal. Time			☐ ✓✓	☐ ✓✓
Delay Start			☐ ✓✓	
Samp. Period			☐ ✓✓	
Pump Period			☐ ✓✓	☐ ✓✓
Total Sample t		mins.		☐ ✓✓
Replicate				
Sample ID	27 -	27 -	☐ ✓✓	☐ ✓✓
Sample Series	HX-	HX -	☐ ✓✓	☐ ✓✓
Flow	mL/min	mL/min	☐ ✓✓	☐ ✓✓
Blank/Spike				
Sample ID	27 -	27 -	☐ ✓✓	☐ ✓✓
Sample Series	HX-	HX -	☐ ✓✓	☐ ✓✓

☐ Pump = 2 Lpm ± 5%

Figure 3. Relative Timing of Active VOC Sampling (page 1 of 3)

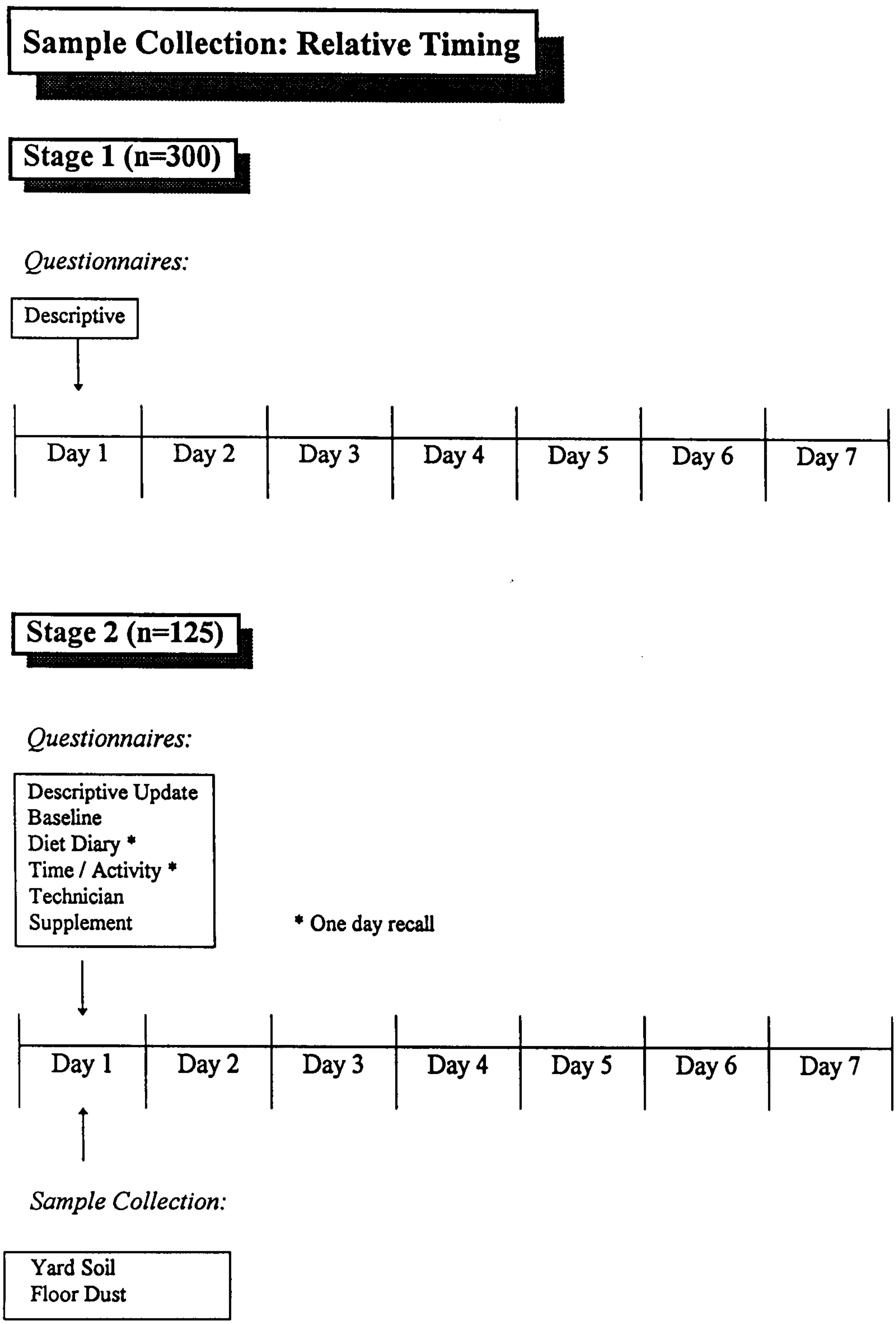
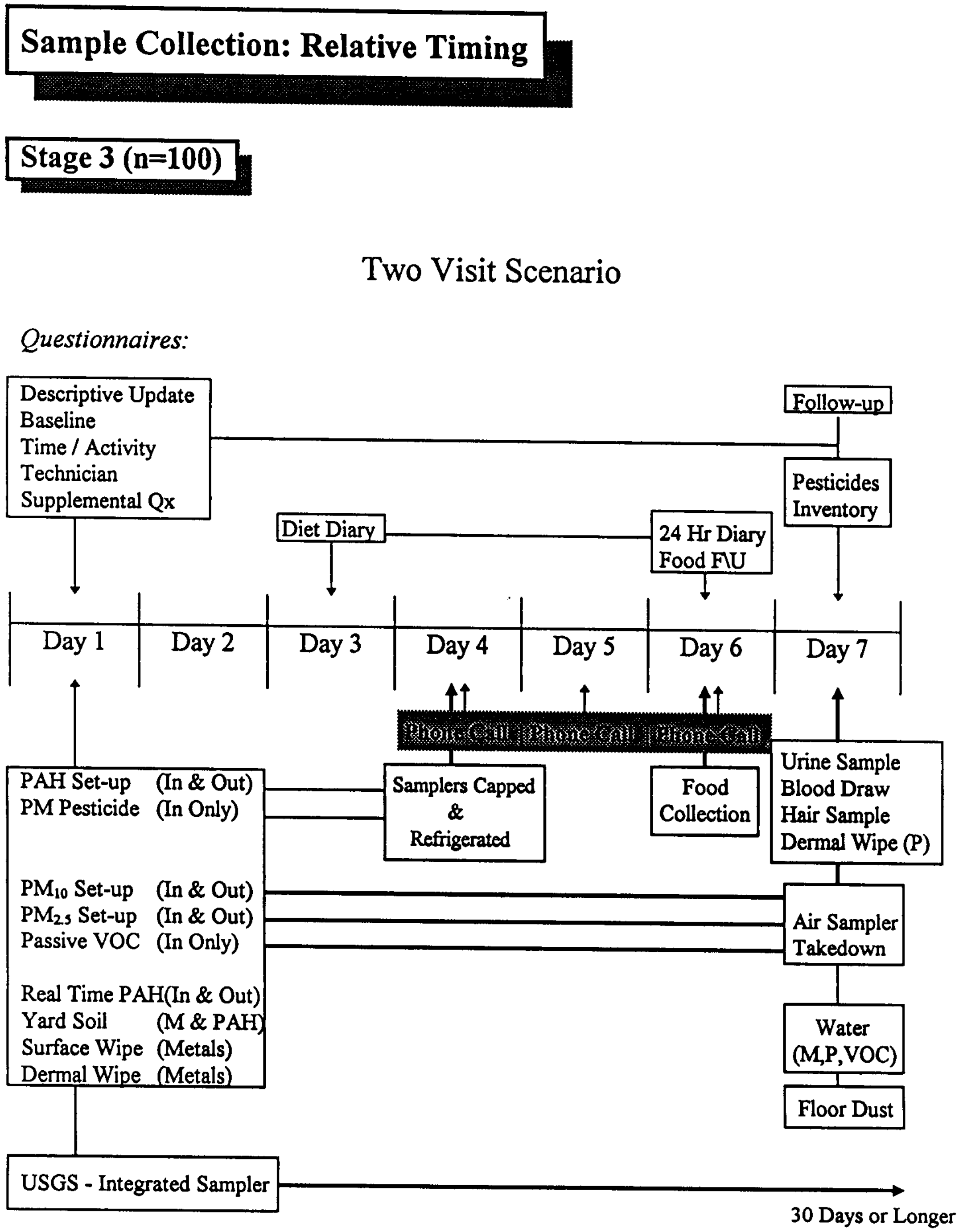


Figure 3. **Relative Timing of Active VOC Sampling (page 2 of 3)**



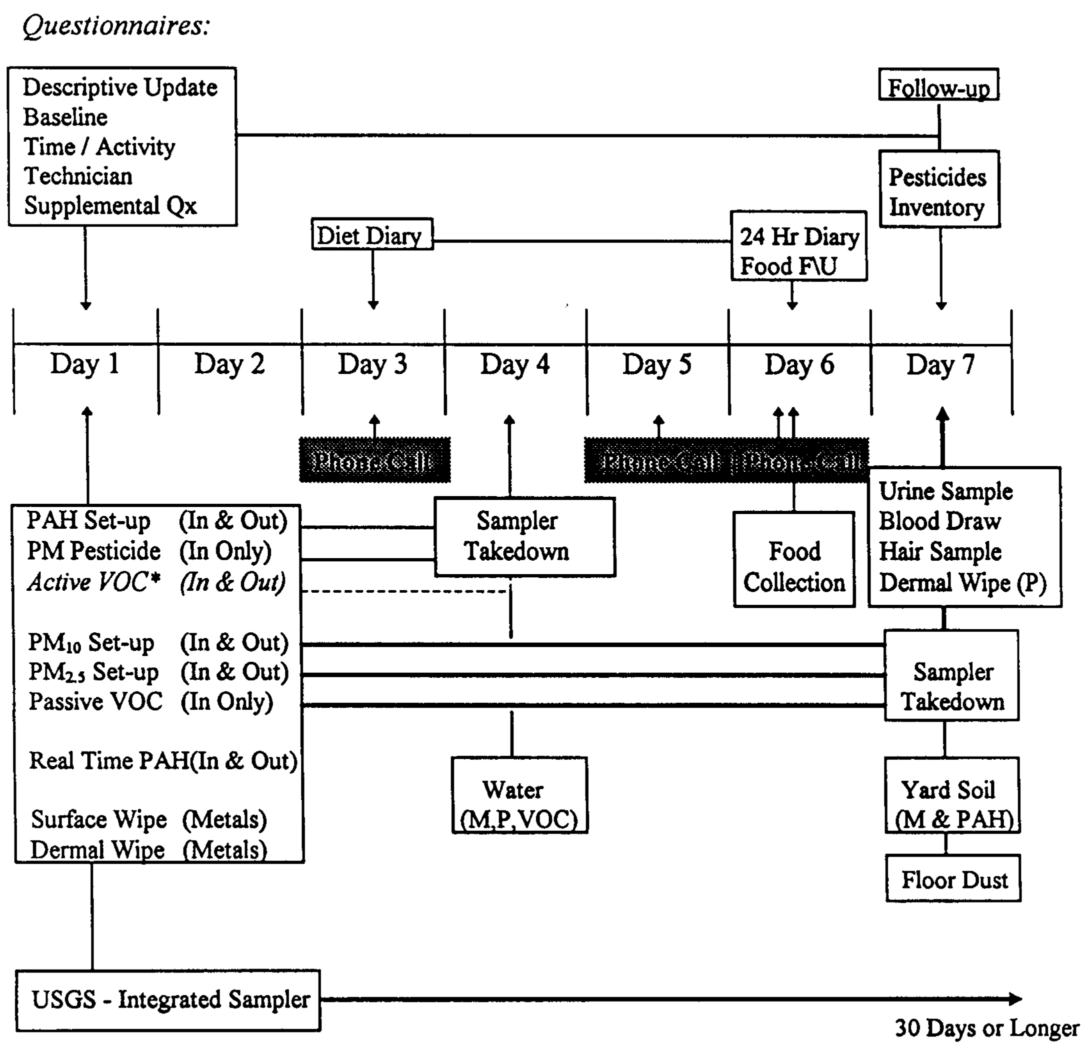
* Active VOC is collected in a subset of 25 homes only

Figure 3. Relative Timing of Active VOC Sampling (page 3 of 3)

Sample Collection: Relative Timing

Stage 3 (n=100)

Three Visit Scenario



* Active VOC is collected in a subset of 25 homes only

Figure 4. SKC Pump Log

SKC Pump Log

Pump ID#:______________ Pump Serial#:______________ Charger ID#: ______________

CHARGING INFO

Batteries Drained Y/N	Total Pump Elapsed Time	Total Pump Run Time	Start Date	Start Time	Stop Date	Stop Time	Recalibrated Y/N	Initials	Comments
[ ] [ ]							[ ] [ ]		
[ ] [ ]							[ ] [ ]		
[ ] [ ]							[ ] [ ]		
[ ] [ ]							[ ] [ ]		
[ ] [ ]							[ ] [ ]		
[ ] [ ]							[ ] [ ]		
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[ ] [ ]							[ ] [ ]		
[ ] [ ]							[ ] [ ]		
[ ] [ ]							[ ] [ ]		

N:\ROGAN\FIELDSAM\SKCLOG

Figure 5. Low Flow Controller and Pump Check Log

PRE-FIELD LOW FLOW CONTROLLER AND PUMP CHECK LOG

CONTROLLER ID#______________________
SKC PUMP ID #______________________
TEST CARBO TRAP ID#__________________
PUMP CONTROLLER RED FLAGGED? Y__ OR N___
If yes, describe problem and resolution:________________________
DIFFUSION CAP ID#____________________

FLOW CHECKS

	DATE	TIME	FLOW METER ID	FLOW RATE	COMMENTS
test 1		:		ml/min	
test 2		:		ml/min	
test 3		:		ml/min	
test 4		:		ml/min	
test 5		:		ml/min	
test 6		:		ml/min	
test 7		:		ml/min	
test 8		:		ml/min	
test 9		:		ml/min	
test 10		:		ml/min	
test 11		:		ml/min	
test 12		:		ml/min	

Average Flow Rate = ______________ml/min

Figure 6. **Active VOC Sampling Troubleshooting Guide**

1) Be very careful not to bend or crimp the carbotraps as this will alter the sample flow.

2) Carefully inspect the diffusion limiting tubing for crimps or bends before leaving for the field. If tubing is crimped, replace before using in the field.

3) Field set-up for active voc sampling flow checks should be as follows:

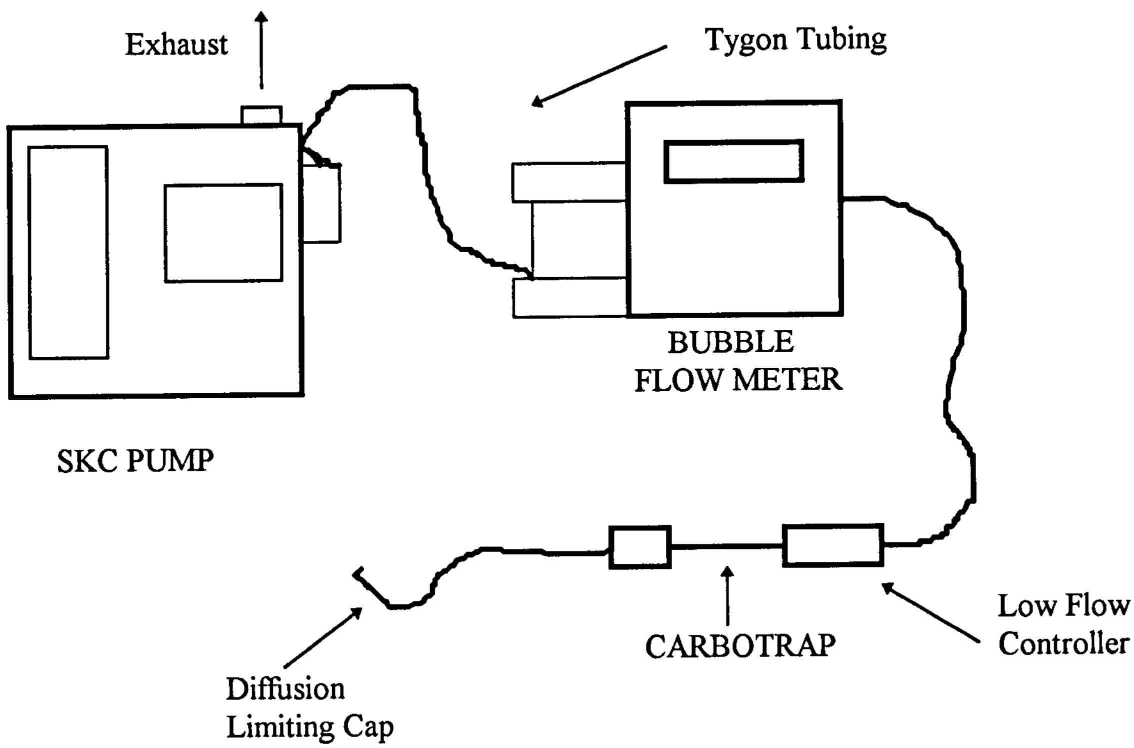

Figure 7. Battelle VOC Sampling Trials (page 1 of 2)

Effect of Diffusion-Limiting Tube and Delay Period on Indoor Air Concentrations of TO-14 Target Compounds (in µg/m³) Collected With Multisorbent Carbotrap Tubes

					Sample period = 72 hr (4320 min) with pump period = 13.33 hr (800 min) @ 5 cm³/min			
		Unexposed Blank	72-Hour delay with diffusion-limiting tube attached		Single pump – single flow controller		Single pump – dual flow controller	
	Compound	HX1022	HX1003	HX1004	HX1006	HX1007*	HX1008	HX1009
1.	dichlorodifluoromethane	1.52	0.40	0.78	0.12		0.13	0.12
2.	methyl chloride	0.00	0.03	0.00	0.03		0.06	0.04
3.	1,2-dichloro-1,1,2,2-tetrafluoroethane	0.00	0.00	0.00	0.00		0.00	0.00
4.	vinyl chloride	0.00	0.00	0.00	0.00		0.00	0.00
[redacted]								
6.	methyl bromide	0.00	0.00	0.00	0.00		0.00	0.00
7.	ethyl chloride	0.00	0.00	0.00	0.44	0.26	0.36	0.34
8.	trichlorofluoromethane	0.00	0.38	0.53	9.23		8.09	8.36
9.	1,1-dichloroethene	0.00	0.00	0.00	0.22	0.20	0.23	0.19
10.	dichloromethane	0.00	0.47	0.60	44.9	31.7	24.0	25.9
11	3-chloropropene	0.00	0.00	0.00	2.99		2.58	2.32
12.	1,1,2-trichloro-1,2,2-trifluoro-ethane	0.00	0.35	0.54	14.4	15.9	12.8	11.7
13.	1,1-dichloroethane	0.00	0.00	0.00	5.46	5.79	4.69	4.41
14.	cis-1,2-dichloroethene	0.00	0.00	0.00	0.11	0.13	0.10	0.09
15.	trichloromethane	0.00	0.08	0.13	9.41	10.5	8.51	7.80
16.	1,2-dichloroethane	0.00	0.00	0.00	15.6	16.1	13.6	13.4
17.	1,1,1-trichloroethane	0.00	0.44	0.59	43.8	48.1	38.1	35.3
[redacted]								

Figure 7. Battelle VOC Sampling Trials (page 2 of 2)

Effect of Diffusion-Limiting Tube and Delay Period on Indoor Air Concentrations of TO-14 Target Compounds (in µg/m^3) Collected With Multisorbent Carbotrap Tubes

		Unexposed Blank	72-Hour delay with diffusion-limiting tube attached		Sample period = 72 hr (4320 min) with pump period = 13.33 hr (800 min) @ 5 cm^3/min: Single pump -- single flow controller		Single pump -- dual flow controller	
	Compound	HX1022	HX1003	HX1004	HX1006	HX1007*	HX1008	HX1009
[redacted]								
19.	carbon tetrachloride	0.00	0.00	0.00	8.70	9.48	9.20	8.69
20.	1,2-dichloropropane	0.00	0.00	0.00	1.00	0.97	0.94	1.10
[redacted]								
22.	cis-1,3-dichloropropene	0.00	0.00	0.00	0.84	0.91	0.77	0.72
23.	trans-1,3-dichloropropene	0.00	0.00	0.00	0.62	0.59	0.56	0.52
24.	1,1,2-trichloroethane	0.00	0.00	0.00	0.33	0.38	0.34	0.36
[redacted]								
26.	1,2-dibromoethane	0.00	0.00	0.00	4.27	3.61	3.47	5.39
27.	tetrachloroethene	0.00	0.00	0.00	1.40	1.27	1.34	1.44
28.	chlorobenzene	0.00	0.00	0.00	0.78	0.56	0.69	0.85
29.	ethylbenzene	0.00	0.00	0.00	1.85	1.10	0.93	1.81
30.	m+p-xylene	0.00	0.00	0.10	2.72	1.00	1.07	2.58
31.	styrene	0.00	0.00	0.00	0.18		0.07	0.19
32.	1,1,2,2-tetrachloroethane	0.00	0.00	0.00	0.61		0.58	0.67
33.	o-xylene	0.00	0.00	0.00	0.83		0.28	0.75
34.	4-ethyl toluene	0.00	0.00	0.00	0.00		0.00	0.00
35.	1,3,5-trimethylbenzene	0.00	0.00	0.00	0.00		0.00	0.00
36.	1,2,4-trimethylbenzene	0.00	0.00	0.00	0.00		0.00	0.00
37	benzyl chloride	0.00	0.00	0.00	0.40		0.13	0.36
38.	m-dichlorobenzene	0.00	0.00	0.00	0.27		0.08	0.27
39.	p-dichlorobenzene	0.00	0.00	0.00	0.22		0.08	0.22
40.	o-dichlorobenzene	0.00	0.00	0.00	0.00		0.00	0.00
41.	1,2,4-trichlorobenzene	0.00	0.00	0.00	0.00		0.00	0.00
42.	hexachlorobutadiene	0.00	0.00	0.00	0.00		0.00	0.00

* No MSD measurements available due to computer problem. Concentrations measured from FID response.