National Human Exposure Assessment Survey (NHEXAS)

Arizona Study

Quality Systems and Implementation Plan for Human Exposure Assessment

The University of Arizona
Tucson, Arizona 85721

Cooperative Agreement CR 821560

Standard Operating Procedure **SOP-UA-F-9.1**

Title: Collection of Dermal Wipe Samples for Pesticides and Metals Analysis

Source: The University of Arizona

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

Collection of Dermal Wipe Samples for Pesticides and Metals Analysis

1.0 PURPOSE AND APPLICABILITY

This standard operating procedure (SOP) describes the procedures for collecting a dermal wipe sample from a participant's hands for the measurement of pesticide or metal residues on the skin. This procedure covers the preparation of the dermal wipe material and field activities. This protocol must be followed to insure consistent data retrieval of dust analyte samples for the NHEXAS Arizona project, AZ Border project (BORDER AZ), and other Health and Environment projects.

2.0 DEFINITIONS

2.1 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.

2.2 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.

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

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

2.5 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.

2.6 FIELD KIT = A sampling tool-box containing appropriate collection and storage utensils. For dermal wipe collection, the kit contains extra polyethylene Ziploc bags (quart and sandwich sizes), disposable non-powdered latex gloves, extra polyethylene pipettes, a psychrometer or hygrometer, three charged **D** cell batteries and extra copies of the Dermal Wipe Sampling Sheet (Fig. 2).

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

2.8 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.

2.9 **HOUSEHOLD**(HH) = The residence occupied by study respondent(s).

2.10 **HOUSEHOLD IDENTIFICATION** 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.

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

2.12 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 Dermal Wipe a unique sample ID number upon receipt from Battelle.

2.13 MUFFLED GLASSWARE = A heat cleaning procedure for glassware to remove residual organic contamination. Glassware is heated in a "muffle" furnace to 400°C for 4 hrs.

2.14 N/A = Not Applicable.

2.15 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.

2.16 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.

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

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

2.19 RESPONDENT = A person in the study population of NHEXAS Arizona, AZ Border project, and 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.

2.20 SAMPLE = The deposit left on the wipe after dermal sampling is complete.

2.21 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.

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

2.23 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.

2.24 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.

2.25 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 D. Camann, "Comparison of Dislodgeable Residue and Handwipe Methods for Pesticide Transfer from Floors," Presented at NHEXAS Dust and Dermal Workshop, Research Triangle Park, NC, January, 1994.

3.2 R.A. Fenske and C. Lu; "Determination of Handwash Removal Efficiency: Incomplete Removal of the Pesticide Chlorpyrifos from Skin by Standard Handwash Techniques" J Am Ind Hyg Assoc; 55, 1994.

4.0 DISCUSSION

4.1 This method describes the collection of surface residues of pesticides or metals from the hands of the primary respondent or participant using SOF-WICK gauze pads. The participant's hands may be sampled for both metals and pesticide analytes. Metals samples are collected first and pesticides are collected several days later on a subsequent visit. Prior to wiping, the gauze is prepared with either distilled water (in the case of a metals sample), or isopropanol (for pesticide sampling). The participant wipes both hands with one gauze pad, and then wipes a second time with a second gauze pad of the same type. Both wipes are combined into a single bag for storage, extraction, and analysis. Be sure

to label the bags and indicate whether the contents are to be analyzed for either metals or pesticide sampling.

4.2 The wipe method selected here for pesticides was shown to provide 97% removal efficiency for chlorpyrifos when 4 mg quantities were applied and then wiped for removal within 1 hr (see 3.1 above). Experiments conducted with wipe solutions of ethanol and isopropanol/water provided much lower wipe removal efficiencies- 20 to 40%- even when much larger doses were applied (up to 1600 mg) and time between application and wipe was negligible (see 3.2 above).

4.3 Dermal wipe gauze pads and solution may be stored at room temperature for up to six months after the date of preparation before they must be returned to Battelle. Dermal wipes must be returned to Battelle within six weeks of sample collection. Post collection storage for pesticide wipes must be -20°C. Dermal wipe samples will be returned for analysis on blue ice.

4.4 The data/sample flow diagram for Dermal Wipe Samples is displayed in Fig. 3. The relative timing of Dermal Wipe Sampling to other sampling by stage is displayed in Fig. 4.

5.0 RESPONSIBILITIES

5.1 Battelle personnel are responsible for:

(a) purchasing wipe material,
(b) cleanup of unexposed wipes before shipment for field use,
(c) shipping wipe materials in pre-packaged units to the HRP Office for use in household sampling,
(d) preparation of the moistening solution and aliquotting into individual vials for field use,
(e) shipping vials to the HRP Office,
(f) assuring that blank levels for each batch of wipes meet acceptability requirements prior to shipping to the HRP Office,
(g) documenting blank levels in a NHEXAS (or any other study) laboratory notebook.

5.2 The Materials Technician is responsible for:

(a) receipt of wipes and vials shipped by Battelle personnel,
(b) logging the material into the Tracking System and generating a unique sample ID for each wipe packet and moistening solution vial in accordance with SOP UA-G-5.X,
(c) storing the wipes and moistening solution at room temperature in the Dermal Wipe Sampling Pre-Field Storage Bin before assignment to the HH,
(d) randomly selecting 10% of each batch of wipes and moistening vials to serve as

UA Lab and Field Blanks,

(e) assigning blanks and samplers to the field and documenting the assignment in the Tracking System,

(f) shipping the exposed samplers and blanks on dry 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 surface wipe collection in the field.

(c) 100% QA check of all Surface Wipe Sampling Sheets within 24 hours of submission by the Team Leader

5.4 The Team Leader is responsible for:

(a) participant and 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 REAGENTS

6.1 Materials

a) Teflon-lined screw-cap vials (10 mL).
b) Polyethylene transfer pipettes (4 mL).
c) SOF-WICK gauze pads (4" x 4" -6 ply; Johnson & Johnson).
d) Polyethylene Ziploc bags, 4 mils thick (4"x4" and quart size).
e) Disposable non-sterile, non-powered latex gloves.
f) Soxhlet extractor (large size, e.g.Kontes 585000-0023; including extractor body, condenser, 300 mL round bottom flask)
g) Heating mantle
h) Variac
i) Boiling chips (Hengar crystals)
j) Stainless steel tongs
k) Teflon tweezers
l) Polyethylene vials (10 mL)
m) Psychrometer, RH Slide Rule and 3 AA batteries, or digital hygrometer
n) Polyethylene Gloves and Kim Wipes.

6.2 Reagents

a) Methylene Chloride (high purity)
b) Distilled deionized water
c) Isopropanol (OPTIMA)

7.0 PROCEDURE

7.1 The pre-extraction of Sof-Wick Gauze Pads (hereafter referred to as wipes) occurs at Battelle.

7.1.1 Load 8 wipes into a Soxhlet extractor. Add 250 mL of methylene chloride to the 300 mL round bottom flask; add a few boiling chips. Connect the condenser. Add the heating mantle and the Variac. Prepare in triplicate for one batch of 24 wipes (either pesticides or metals).

7.1.2 Continue with Soxhlet extraction as described in BCO SOP-L-2.X. Extract wipes overnight (14-16 hrs).

7.1.3 At the end of extraction, drain residual methylene chloride from the Soxhlet extractor and remove wipes to a clean dry mat of Kim-Wipes in a laboratory hood using methylene chloride-rinsed tongs. Cover the wipes with a single layer of Kim-Wipes.

7.1.4 Allow wipes to dry for approximately 10 min.

7.1.5 Using methylene chloride-rinsed tweezers, place two dry wipes in a Zip-lock (4"x4") bag. Place 12 sample bags into a larger Zip-lock bag. Ship in this way to the field.

7.1.6 Aliquot 8 mL volumes of isopropanol into 10 mL glass vials. Label as moistening solvent (isopropanol) for pesticide hand wipes. Send at least 26 vials per batch of hand wipes.

7.1.7 Aliquot 8 mL volumes of distilled deionized water into 10 mL polyethylene vials. Label as moistening solvent (water) for metals hand wipes. Send at least 26 vials per batch of hand wipes.

7.2 Field Hand Wipe Procedure

Wipes are taken into the field at room temperature in the HH bucket. After collection, the wipes are returned to the Field Office on blue-ice. The wipes are stored at -20^{o}C pending shipment to Battelle on blue ice.

7.2.1 The hands of the primary respondent are wiped for metals during visit one of Stage III

sampling. The pesticide dermal wipe sample is also collected from the hands of the primary respondent on visit three Stage III. If only one wipe may be collected, the metals wipe has priority.

7.2.2 The Field Team Member puts on a pair of latex gloves and removes both gauze wipes from the Ziploc bag and places them on a kim-wipe on a clean surface.

7.2.3 The Team Member locates either a vial of isopropanol or a vial of water, opens the vial and draws up half of the 8 mL volume with a disposable pipette. The vials are marked at the 4 mL level.

7.2.4 The Team Member instructs the respondent to remove any watches or bracelets before sampling. If the respondent is wearing any rings or other jewelry on their fingers, instruct them not to remove the rings. Note that the respondent was wearing rings when the sample was taken on the Field Sheet. Watches and bracelets are removed before sampling. Rings and finger ornamentation are not removed as many respondents will not be physically able to remove their rings. Note discrepancies and observations on the field sheet.

7.2.5 The Team Member asks the respondent to pick up one of the gauze wipes and leave the second on the kim-wipe. The respondent is instructed to 'cup' his/her hands together in a bowl shape and place the wipe flat on his/her palms. The Team Member then wets the gauze thoroughly with the 4 mL solution.

7.2.6 The participant is instructed to wipe both sides of each hand thoroughly with the gauze wipe including their fingers. The participant is told not to wipe above the wrist.

7.2.7 While the respondent is wiping his/her hands complete the appropriate fields on the Field Data Sheet by asking about the last time the respondent washed his/her hands, or whether he/she is wearing nail-polish, etc.

7.2.8 Allow sufficient time for the respondent to thoroughly wipe his/her hands. This process should take no less than one minute. When complete, the respondent places the gauze wipe into the original Ziploc bag (which the Team Leader holds open). The Team Leader does not touch the gauze pad once the respondent has wiped his/her hands.

7.2.9 Repeat Steps 7.2.5-7.2.8, using the second wipe and the remainder of the solution in the selected vial. The participant is instructed to be more careful to wipe the fingers and the area between the fingers with this second wipe. This second gauze wipe is added to the same Ziploc bag as the first wipe.

7.2.10 Seal the bag and label it with the sample identification number, the HHID, IRN, date, and whether it is a metal or pesticide wipe.

7.2.11 Determine the glove-size of the respondent using a glove sizing chart after sampling is complete and record the size on the Field Sheet.

7.2.12 Place the Ziploc bag containing the sample in a cooler on blue ice packs, until it is returned to the Field Office.

7.2.13 Repeat the procedure for the other analyte class during the next visit (72 hours) or on the last visit (7 days).

7.2.14 Store samples at -20°C until shipment (on blue ice) by Federal Express to Battelle for analysis. Samples must be forwarded to Battelle within six weeks of collection or the sample integrity may be compromised. Shipments to Battelle will be made weekly.

7.3 Calculations - N/A

7.4 Quality Control : Ten percent of all samples will be used for QA/QC purposes.

7.4.1 To achieve the necessary levels of QA/QC samples, the following set of analyses are planned for each batch (either pesticides or metals) of 24 wipes that are prepared:

18 wipes	hand wipes of 9 people (pesticides only or metals only)
2 wipes	1 pre-shipment analysis for materials suitability
2 wipes	1 field spike
2 wipes	1 field blank

This batch will be collected for either pesticides or metals hand wipes. There will not be any field duplicates, as that will not be possible for this matrix. There will be 25% overall QA (3 samples out of every 12 hours).

7.4.2 To prepare a field blank sample, prepare two sample wipes as described above. Place the wipes in the Ziploc bag and mark as a field blank. Store and ship as per instructions for actual samples.

7.4.3 For the pesticide field spike sample, prepare a wipe as if for an actual hand wipe sample, and spike with 50 µL of the Pesticide Field Spike solution (0.25 ug/analyte) (see SOP BCO- L-2.X) using a 25 mL syringe. Spike the wipe by dragging the syringe lightly over the center area of the wipe as the solution is expelled. After spiking the wipe, place it in the zip-lock bag. The second wipe is moistened and placed in the same Ziploc bag. Apply spike reagent while wipe is on a Kimwipe.

7.4.4 For the metals field spike sample, prepare a wipe as if for an actual hand wipe sample, but spike with 50 uL of the Metals Field Spike solution using an adjustable micropipette with disposable plastic tip. Spike the wipe as described above and place it in the Ziploc bag.

The second wipe is moistened and placed in the Ziploc bag without any spike. Apply spike reagent while gauze wipe rests on a clean Kimwipe.

7.4.5 Laboratory blanks will not be analyzed concurrently with sample sets unless a problem is found with the field blanks. At such time, laboratory method blanks (lab blanks), trip blanks (a blank that is shipped from Battelle to UA and returned without exposure), and field blanks will be analyzed to pinpoint and correct the source of contamination.

7.4.6 Tolerance Limits

Field tolerance limits in the collection of Dermal Wipe Samples are related to the length of time a respondent has the gauze wipe and reagent in their hands. The Team Member should encourage the respondent to wipe carefully. Each gauze wipe should undergo at least one minute of active sampling. Document samples which are not actively being collected for at least one minute, and those that were sampled for longer than four minutes.

7.4.7 Detection Limits

Estimation is used for elapsed time in the collection of Dermal Wipe Samples. Each gauze wipe should be used by the respondent for one minute +/- 15 seconds. Timers or stop-watches are not necessary. The determination of glove size is used as an approximation of hand size. Glove size determination is limited to the sizing chart used.

7.4.8 Corrective Actions

Apparent mis-labeling 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.

8.1.1 This Record (Fig.1) will serve as the primary record of sample custody after collection in the field. The Team Leader and the collector are responsible for the thorough completion of this form.

8.1.2 The completed original Chain of Custody Record will remain with the data sample at all times.

8.2 Dermal Wipe Sampling Sheet

8.2.1 This record documents sampling location, IRN, age, gender, date, time and any problems

encountered (Fig. 2). The form is photocopied once returned to the field office, and the original is forwarded to Battelle with the samples and Chain of Custody.

8.3 NHEXAS (or any other study) Lab Notebooks

8.3.1 A record of the blank levels of pesticides and metals in each batch of cleaned wipes will be maintained in NHEXAS (or other study) laboratory notebooks set aside for materials preparation information. These notebooks will record the batch number, the date of extraction, the lot number for the wipes, and the lot number of the methylene chloride used for extraction. These notebooks (one for metals and one for pesticides) will be retained in the respective extraction laboratories until the conclusion of the program, and will serve as a continuing file on the expected performance of the cleanup step. At the conclusion of the program these notebooks will be transferred to the Principal Investigators' office at Battelle.

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]		___/___/___	___:___	
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[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. Dermal Wipe Sampling Data Sheet

DERMAL WIPE SAMPLING SHEET

Project = NHEXAS AZ Stage # _____ HHID = _____/____

Form Id = UA-F9.0-1.0 Team Leader = _____ Date ____/____/____ (mo / day / yr)

[ ] Pesticide Dermal Wipe

[ ] Metals Dermal Wipe

SAMPLE ID _____

IRN _____ Age _____ Gender _____

Both wipes sampled for at least one minute each?
[Y] [N]

WB_____
DB_____
RH_____

Comments:_____

[Y] [N] Wearing Rings? If yes, indicate material _____
[Y] [N] Nail Polish?

GLOVESIZE (from chart) = _____

Comments:_____

*

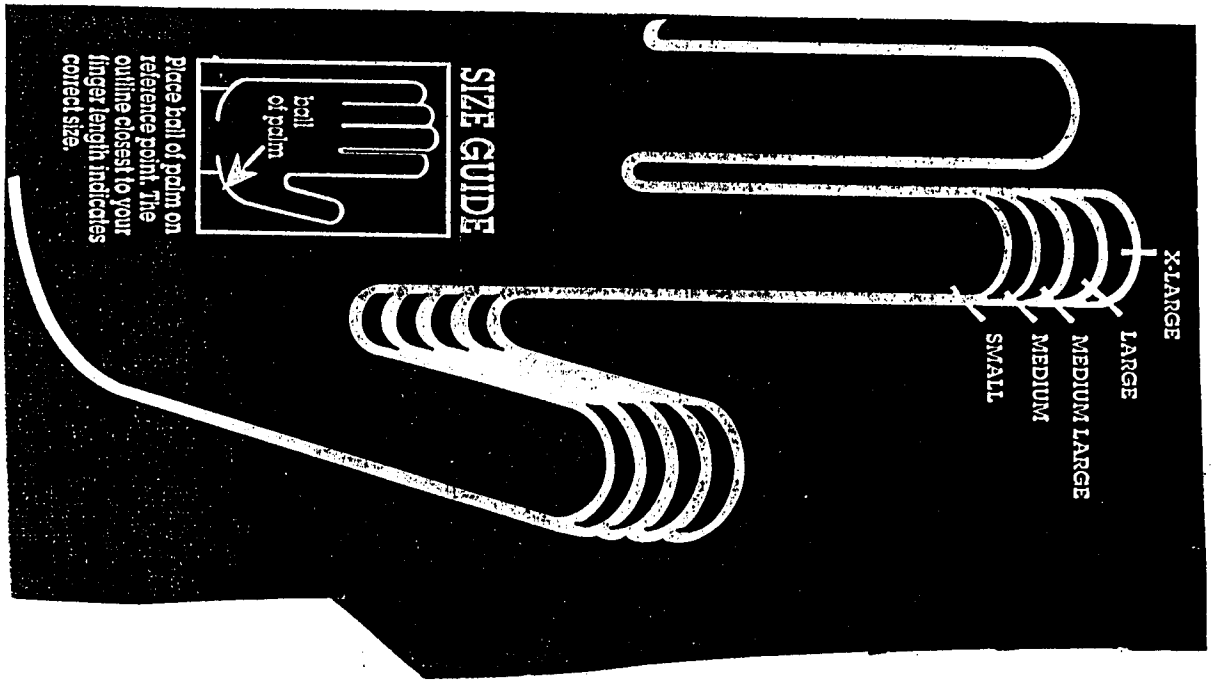


Figure 3. Data/Sample Flow Diagram for Dermal Wipe Samples

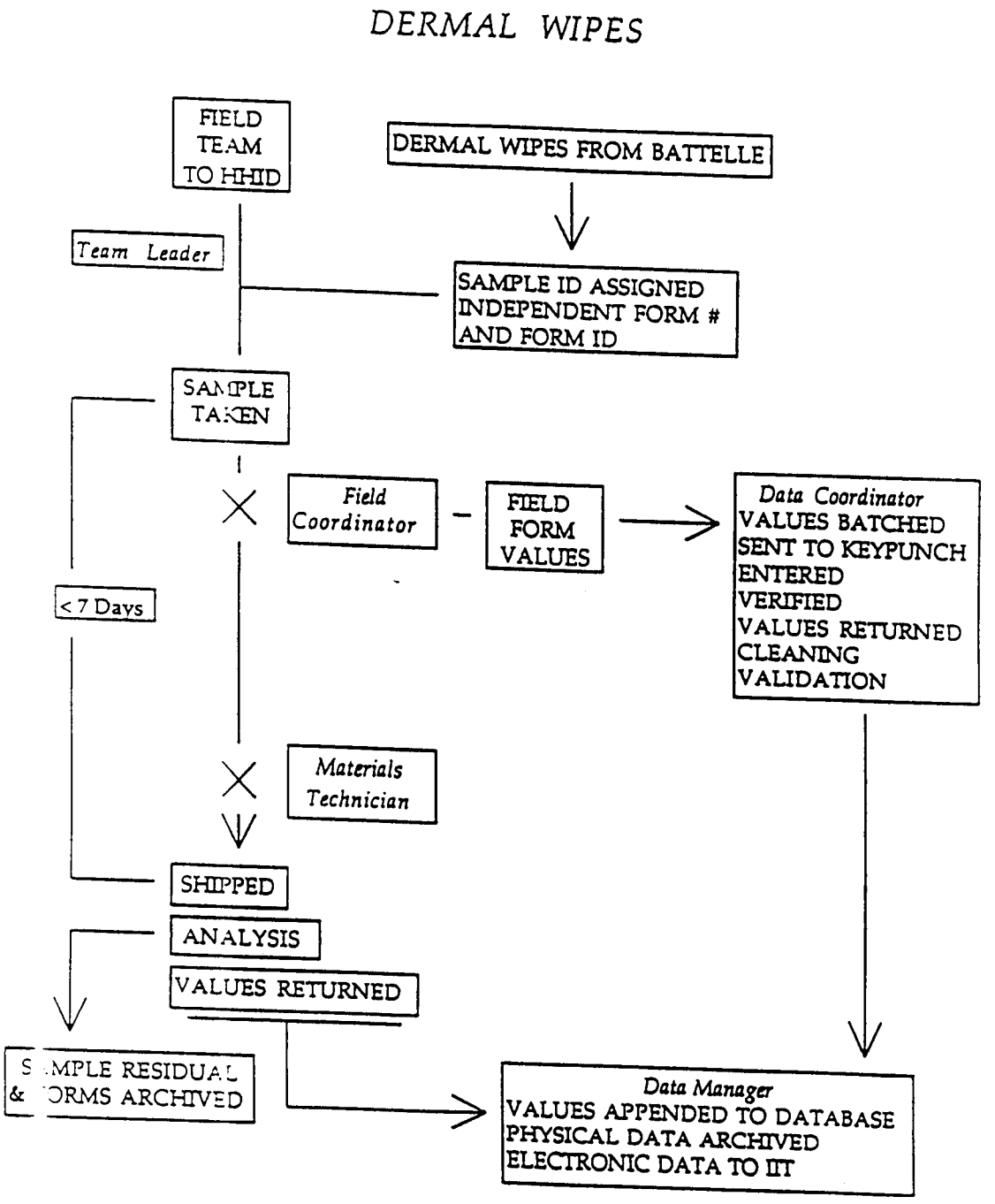


Figure 4. Relative Timing of Sample Collection by Stage (page 1 of 3)

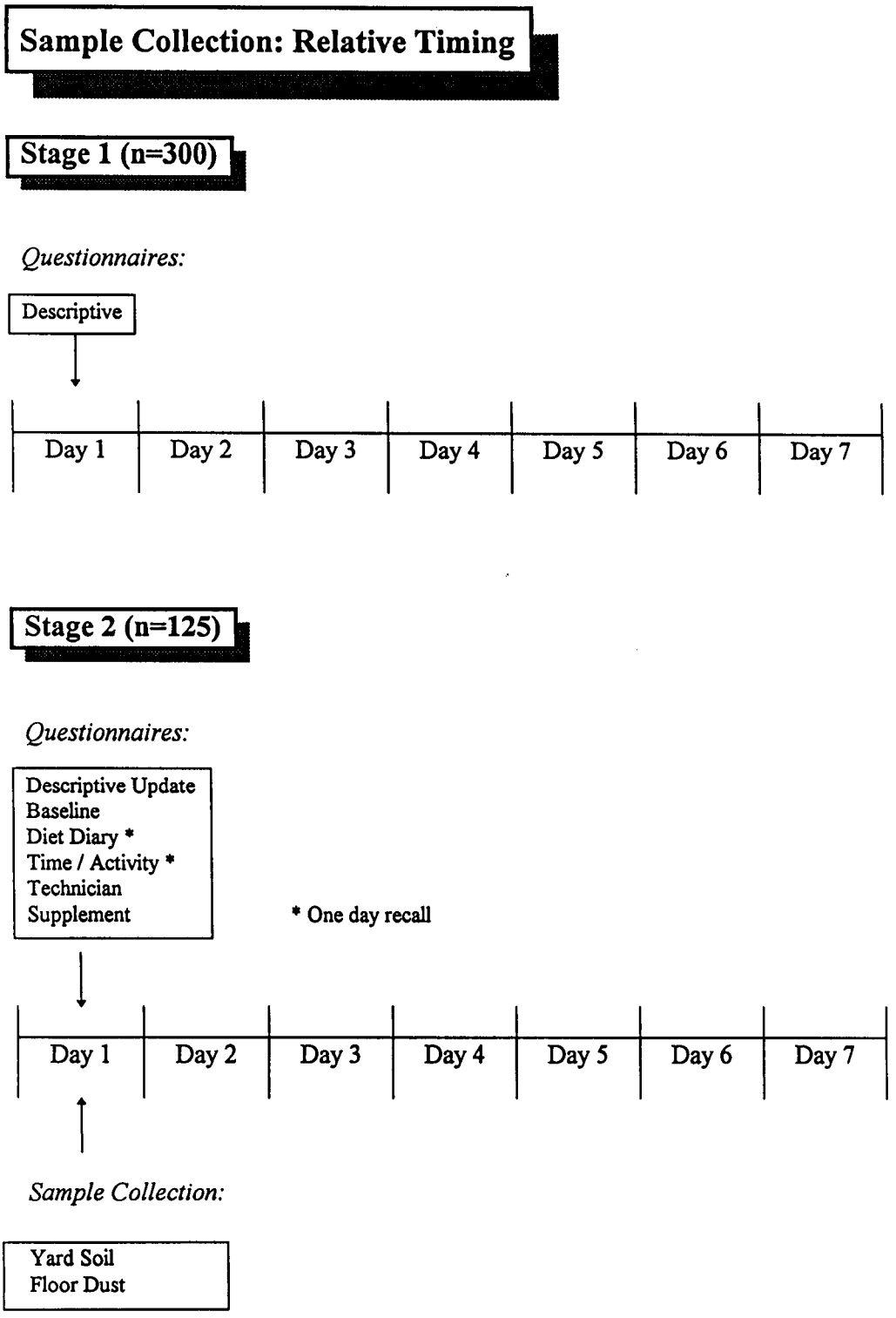
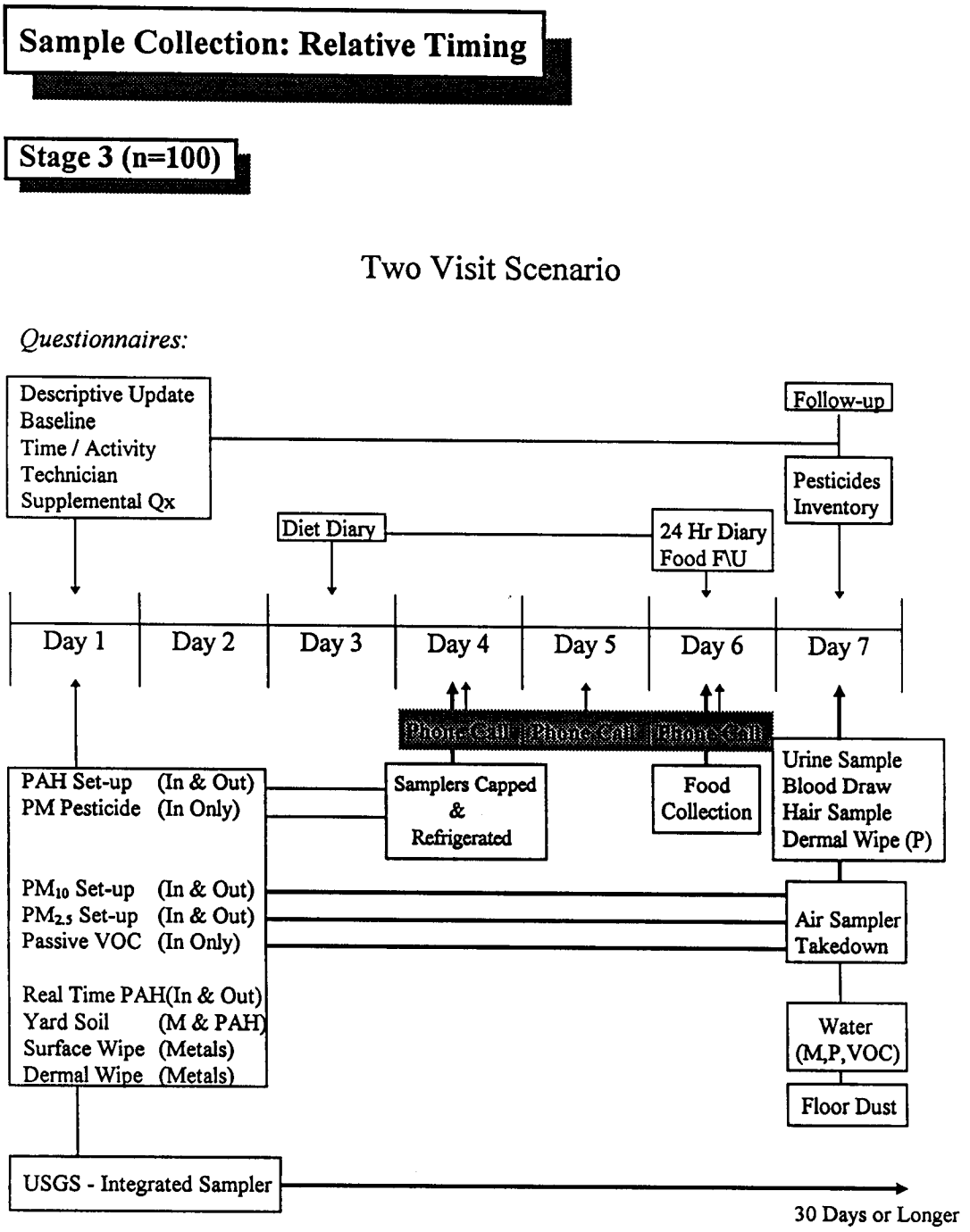
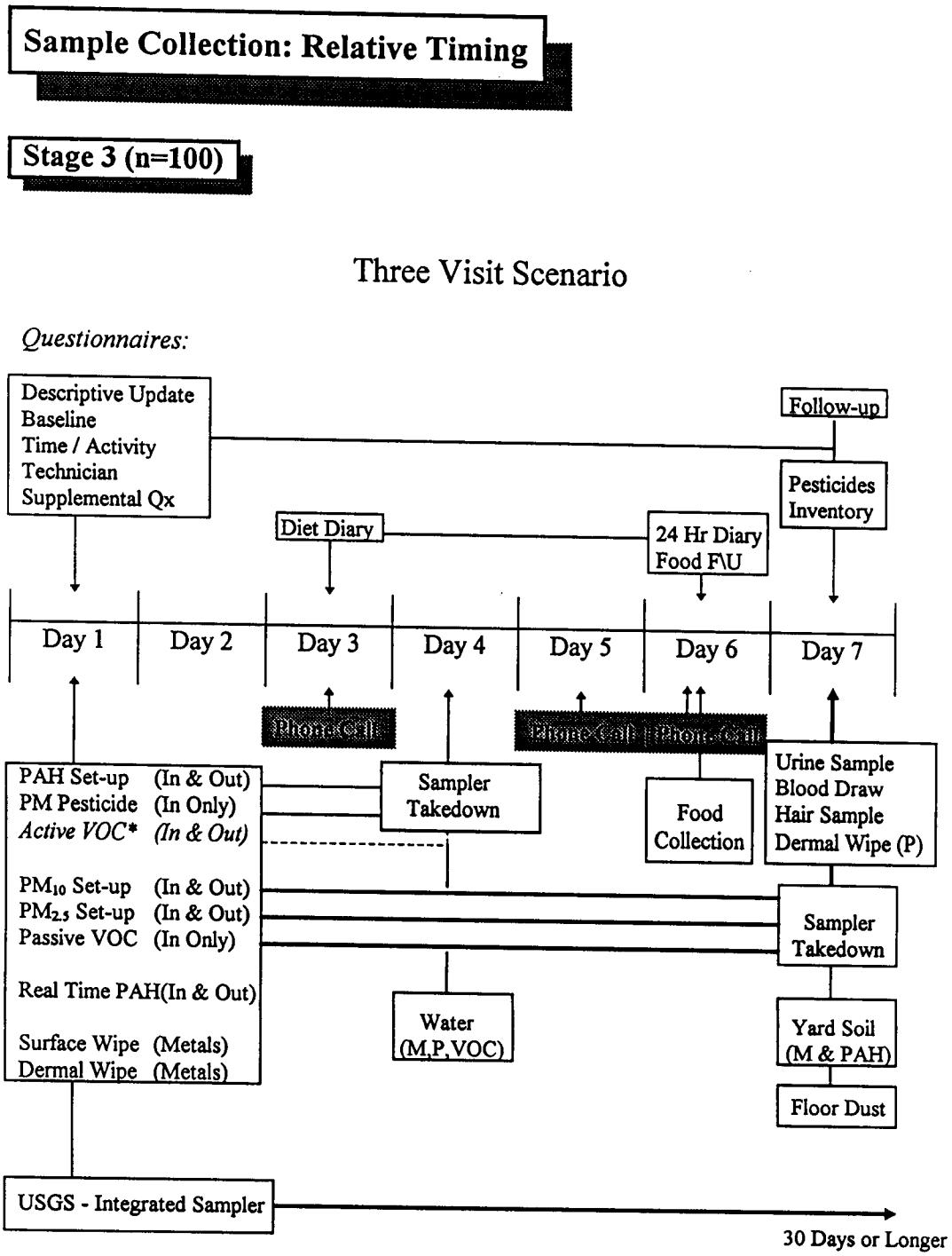


Figure 4. Relative Timing of Sample Collection by Stage (page 2 of 3)



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

Figure 4. Relative Timing of Sample Collection by Stage (page 3 of 3)



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

Figure 5. Field Notes and Trouble Shooting Guide for Dermal Wipe Sample Collection

1. If only one dermal wipe may be collected, collect the metals wipe.
2. Note any missing digits or other unusual occurrences on the dermal wipe sampling sheet.
3. Warn the respondent that the isopropanol reagent used to collect the pesticide dermal wipe may remove nail polish, or may sting if the respondent has any open wounds on his/her hands.