

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-15.1

Title: Field Collection of Food and Beverage Samples

Source: The University of Arizona

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

Notice: The U.S. Environmental Protection Agency (EPA), through its Office of Research and Development (ORD), partially funded and collaborated in the research described here. This protocol is part of the Quality Systems Implementation Plan (QSIP) that was reviewed by the EPA and approved for use in this demonstration/scoping study. Mention of trade names or commercial products does not constitute endorsement or recommendation by EPA for use.

Field Collection of Food and Beverage Samples

1.0 PURPOSE AND APPLICABILITY

This SOP provides a uniform procedure for the collection of a complete (all meals, snacks and beverages), one-day (24 hour) duplicate diet sample from the primary respondent. Participants will keep a duplicate portion of all food consumed in a 24 hour period. Samples saved for analysis will be identical by type and amount to those portions consumed by the respondent. This procedure must be followed to ensure consistent data retrieval of metal and pesticide analyte samples for the NHEXAS Arizona project, AZ Border project, 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 BACK-UP FIELD KIT = Each field vehicle carries a box containing back-up forms in case of field implementation problems.
- 2.3 BUCKET = A plastic container with a buckle top. One bucket is assigned to each household to be visited. Household identification and stage numbers are listed on the outside of the container. The bucket contains all paperwork to be completed by field staff or household respondents. It serves as the primary vehicle for securing and transporting forms, and data from the field through the course of the study.
- 2.4 CHAIN OF CUSTODY RECORD = A vital data tracking and quality assurance form which is attached to the food sampling data sheet. Chain of custody commences with sample collection by Field Team Members (fig 3).
- 2.5 COOLER = One of three alternative methods for keeping collected foods at 4°C during and after collection until shipment. "Cooler" may be a 40 quart cooler with reusable ice packs, an electric cooler, or storage in the primary respondent's refrigerator. The choice of cooling agent (cooler) will be determined by the Team Leader as appropriate.
- 2.6 DUPLICATE SAMPLE = A duplicate portion of food consumed by the primary respondent. Duplicate portions will be estimated visually by the respondent and will be approximately identical to the portion consumed in terms of preparation method, ingredients and quantity.

- 2.7 FOOD COLLECTION KIT = A sampling tool-box containing appropriate collection and storage utensils. For food collection the kit will supply solid and liquid food containers, indelible labeling pen, and additional copies of the 24 Hour Food Diary (Fig. 1), the Food Diary Follow-up (Fig. 2), and the Food Collection Data Sheet and Chain of Custody Record (Fig. 3).
- 2.8 FOOD DIARY FOLLOW-UP = A questionnaire reviewing various components of the 24 Hour Food Diary (Fig. 2).
- 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) associated with a study household.
- 2.11 LIQUID FOOD CONTAINER = A high density polyethylene plastic screw-top container. It is used for the collection of the liquid portion of the 24 hour composite food sample.
- 2.12 MEAL = Any foods consumed by the respondent at a single "sitting". "Meal" includes breakfast, lunch, dinner and snacks, regardless of the quantity consumed.
- 2.13 N/A = Not Applicable.
- 2.14 NHEXAS Arizona = Acronym for National Exposure Assessment Survey, a research project conducted in Arizona by the University of Arizona / Battelle / Illinois Institute of Technology Consortium.
- 2.15 PACKET = This is a large envelope which holds the physical "hard copy" questionnaires and field sampling forms collected from a specific study household.
- 2.16 SOLID FOOD CONTAINER = A polyethylene resealable container. It is used for the collection of the solid portion of the one-day composite sample. Meals are collected in individual freezer Ziploc bags, sealed, and stored in the solid food containers.
- 2.17 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.
- 2.18 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.

2.19 TWENTY FOUR (24) HOUR FOOD DIARY = A daily data tracking sheet completed by the respondent and used to record specific information regarding food samples collected and stored for the one day dietary composite sample (see Fig.1).

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

3.2 RTI/5417/02-03 IG. 1993. *Procedure for collection, storage and shipment of home contact duplicate diet food and beverage samples for metal contaminants* in Guidelines for Measuring Personal Dietary Exposure to Environmental Contaminants.

4.0 DISCUSSION

4.1 This SOP outlines the correct procedure for the collection, storage and transfer of duplicate diet food and duplicate diet beverage samples at participating households according to the strategies outlined in the NHEXAS project, AZ border project and other Health and Environment projects. All foods and beverages consumed regardless of source will be collected. The respondent will collect a sample of all water consumed in a day, except any water from a household source, and add it to the beverage container. Food saved for analysis will be prepared by the respondent and apportioned through visual estimation.

Household water will be evaluated separately according to SOP #UA-F-16.X, SOP #UA-F-17.X and SOP #UA-F-18.X.

4.2 The respondent will store a duplicate sample of every meal in either a solid food or a liquid food container as appropriate. The respondent will record each meal ingested and food sample submitted in the 24 Hour Food Diary (Fig.1). A composite sample of food ingested over a period of one day will be collected.

4.3 The samples will be processed for testing of metals and pesticides upon return to the Field Office. Samples will be frozen before shipment to FDA. Samples will be shipped with appropriate chain of custody records on blue-ice. Proper sample collection, custody and handling must be of primary concern to all field staff. The flow and custody of food and beverage samples is shown in Figure 5, 'The Handling and Data Flow of Food Samples'.

4.4 The relative timing of food and beverage collection to other sampling types by stage is shown in Fig. 7.

5.0 RESPONSIBILITIES

5.1 The Team Leader is responsible for:

- (a) arranging sampling dates and times with the HH;
- (b) respondent training and questionnaire administration;
- (c) completion of the Chain of Custody Record; and
- (d) quality control checks in the field.

5.2 All Team Members are responsible for:

- (a) proper sample collection;
- (b) properly storing and labeling the collected sample;
- (c) transportation of sample to the Field Office; and
- (d) quality control checks in the field.
- (e) completion of food sampling and handling data sheet (fig. 3)

5.3 The Field Coordinator is responsible for:

- (a) 100% QA check of field forms;
- (b) performing 10% QA in-field audit of collection, transportation and storage methods.

5.4 The Laboratory Supervisor is responsible for:

- (a) receiving the food samples from the Materials Technician and evaluating them according to SOP # UA-L-13.X;
- (b) maintaining the integrity of the samples after collection; and
- (c) informing the Materials Technician when the sample is ready to be shipped to FDA.

5.5 The Materials Technician is responsible for:

- (a) assigning food collection kits to each HH for sampling,
- (b) recording the assigned kits in the NHEXAS Tracking System, and
- (c) shipping the food to FDA in a manner that preserves sample integrity.

6.0 MATERIALS AND REAGENTS

6.1 Materials

Materials required for food collection include :

- (a) solid and liquid food containers,
- (b) Ziploc Freezer Bags,
- (c) labeling pen,
- (d) cooler,
- (e) additional copies of the 24 Hour Food Diary (Fig. 1),
- (f) additional copies of the Food Diary Follow-up (Fig. 2), and
- (g) additional copies of the Food Collection Data Sheet and Chain of Custody Record (Fig. 3).

6.2 Reagents - N/A

7.0 PROCEDURE

7.1 Preparation of Standards and Blanks

- (a) Assembly of equipment:
 - 1) 1 L capacity Panasonic food Processor.
 - 2) 1 L capacity Osterizer blender.
 - 3) Power drill with mixing attachment.
 - 4) Cleaned 30 gallon plastic container.
 - 5) Cleaned 5 gallon plastic container.
 - 6) 1 plastic cup with 750mL capacity.
- (b) The items of food listed in **Appendix A** were bought in numerous grocery stores and restaurants to establish a variety of foods for aliquots.
- (c) Food preparation: All food items were prepared as technicians would choose to eat them.
- (d) The food was shredded in an Panasonic food processor and mixed with other foods and liquids from Appendix A for easier blending.
- (e) The foods were then blended at the highest speed (liquefy) in an Osterizer blender one liter at a time.
- (f) After thorough blending, each liter was poured into the cleaned 30 gallon plastic container.
- (g) The result of steps 7.1 (a) through 7.1 (f) is a food slurry (36.5L). With the addition of each new liter of food slurry, the contents of the large plastic container were mixed using the drill-mixer.

- (h) When all the food was in a thoroughly mixed slurry form, 25 Food Field blank samples were prepared by filling the 750 mL container and pouring the contents into a gallon size ziploc bag. Although the food was weighed in grams, samples are measured in mL. Each mL of slurry weighs approximately 1 gram.
- (i) The bags were laid flat on trays and frozen at -5 to -10°C, where they will remain until use.
- (j) Once frozen, samples were labeled as field food blanks.
- (k) One slurry was made. Blanks were removed first. Then, the remaining slurried food (from 7.1 h.) was placed in the clean 5 gallon container, covered, and refrigerated pending the addition of a freshly prepared food spike matrix.
- (l) The refrigerated slurried food was again thoroughly mixed 68 hours later, when the spiked solutions were added (see 7.1.1).
- (m) Of this mix, 500 mL was taken out to evaluate any potential change in the food slurry in response to the 68 hour delay while refrigerated.
- (n) Safety apparatus including vinyl gloves were worn by technicians.
- (o) The 17.25mL of food slurry was spiked with 3 mL of the prepared metal and pesticide spiking solution. Note: The slurried food used for spiking (17.25 L) is a subset of the total slurry. The other portion was used to make the blanks (18.75 L).
- (p) Steps 7.1(b) through 7.1(j) were repeated.

7.1.1 Reagents -

- (a) The metal spike was prepared at Battelle Memorial Laboratories (Columbus, Ohio: Dr. Sydney Gordon) from standard NIST Chemical Solutions.
- (b) Metal Spike:

Five Nist metal solutions were added to 2% HNO₃ solution. The solution was calibrated for addition to 15L of food. The solution was added to a greater volume of slurry (17.25L). Table 1 shows the theoretical values prepared by Battelle and the actual concentrations found in each spiked sample.
- (c) The pesticide spike was prepared at Battelle Memorial Laboratories (Columbus,

Ohio: Dr. Marcia Nishioka).

(d) Pesticide Spike:

Two pesticide solutions were added to acetone. The solution was calibrated for addition to 15L of food. The solution was added to a greater volume of slurry (17.25L). Table 2 shows the theoretical value calculated by Battelle for 15L of food and the actual concentration expected in the 17.25L of slurry to which the spike was added.

Table 1. Concentration of metals in spike matrix and spiked food samples.
(for the calculations 1 mL of slurry is estimated as weighing 1 gram)

Metal Concentration in Spike Solution	Concentration for 15 L of Food (Battelle Calc.)	Concentration expected in each gram of prepared food spike
Cd 0.1 mg/mL	20 ng/g	17.1 ng/g
As 0.2 mg/mL	40 ng/g	34.2 ng/g
Cr 0.2 mg/mL	40 ng/g	34.2 ng/g
Pb 0.2 mg/mL	40 ng/g	34.2 ng/g
Ni 0.5 mg/mL	100 ng/g	85.5 ng/g

Table 2. Concentration of pesticides in spike matrix and spiked food samples.
(For the purposes of calculation, 1 mL of slurry is estimated as weighing 1 gram)

Pesticide Concentration in Spike Solution provided by Battelle	Concentration for 15 L of Food (Battelle Calc.)	Concentration expected in each gram of prepare food spike
Chlorpyrifos 0.1 mg/mL	20 ng/g	17.1 ng/g
Diazinon 0.1 mg/mL	20 ng/g	34.2 ng/g

7.1.2 Standards & Blanks

- Of the original 36.5L of food slurry, 48 samples of about 750 mL were made. 500 mL remained. 25 samples (18.75L) served as "blanks" and 23 samples (17.25L) were spiked with select metals and pesticides and labeled "spikes".
- Either a blank or a spiked sample is carried into the field, handled and shipped under the same conditions as a 'live sample' in 10% of all homes sampled.

7.1.3 Equipment preparation for Household Food Sampling

Make sure that the Food Collection Kit and HH Bucket are stocked with appropriate materials; specifically: solid and liquid food containers, labeling pen, cooler and additional copies of the 24 Hour Food Diary (Fig. 1), the Food Diary Follow-up (Fig. 2), and the Food Collection Data Sheet and Chain of Custody Record (Fig. 3). The particular copies of these forms assigned to the HH will be in the HH bucket, however, additional copies of each form are in the back-up Field Kit.

7.2 Sample Collection

7.2.1 Food pre-collection instructions.

- (a) Prior to the beginning of the food collection period the respondent will be instructed on how to collect the food and beverage samples according to the instructions in the 'How To Collect Food and Beverages' hand-out (Fig. 4).
- (b) Team Leader will read through the instructions with the respondent and answer any questions as needed.
- (c) Team members will demonstrate the use of the solid and liquid food containers on a set of demonstration containers as necessary.
- (d) Team members will emphasize that the respondent will have to plan ahead to prepare enough food for the duplicate portions of food and beverages.
- (e) Team members will emphasize that the study requires that respondents eat what would usually consume if they were not participating in the study.
- (f) The Team Leader will arrange for financial compensation (see UA-F-23.X) and a follow-up sample collection date.
- (g) The Team will leave the collection containers with the HH after having ensured that the respondents understand the collection process. If the family lacks a refrigerator, a cooler with suitable quantities of ice will be left.

7.2.2 Pre-Collection instructions for 24-Hour Food Diary

- (a) Prior to the beginning of the food collection period the respondent will be instructed on how to collect the food and beverage samples and complete the 24-Hour Food Diary according to the instructions in the diary (Fig. 1).
- (b) Team Leader will read through the instructions with the respondent and answer any questions as needed.

7.2.3 Post Diary Sample Collection

Composite food samples will be picked-up by the Field Team on the day after the respondent has collected their food. The food diary will be reviewed with the respondent, and the Food Diary Follow-up questionnaire will be administered.

- (a) The staff will examine the diary and probe entries recorded by the respondent to determine the diary's completeness.
- (b) For food mixtures listed, the respondent will be asked to describe the primary ingredients and the Team Member will record these as needed.
- (c) The Team Leader will compare items listed in the diary with duplicate samples in the food containers. All discrepancies will resolved in the field.
- (d) The Team Leader will be sure to identify each food sample with the respondent and record supplemental information regarding food identity on the sample containers as appropriate.
- (e) Team Leader will complete portion size, preparation method and source information for each item listed in the diary.

7.2.4 Sample Pickup

- (a) After correlation of diary record with sample container contents and discrepancy resolution, the Team Leader will make sure that all pre-labeled containers have the correct HHID, date and time of collection, and sample ID.
- (b) All samples will be properly sealed and stored in a cooler at 4°C for transport to the Field Office.
- (c) The Food Diary Follow-up questionnaire will be administered to the respondent by the Team Leader.

7.2.5 Sample Storage and Shipment.

- (a) Upon return to the Field Office, the samples will be examined and prepared in the laboratory for shipment (SOP # UA-L-13.X). The contents of the sample will be compared against the entries in the diary by UA Laboratory Staff. Discrepancies will be noted on the 24-Hour Food Diary Check sheet (Fig. 11). The presence or absence of alcohol in the sample will also be noted on the check sheet.
- (b) Prior to shipment the Lab Supervisor will be responsible for sample integrity. The

samples will be processed, frozen and shipped within a week of collection.

- (c) The Materials Technician will complete the NHEXAS AZ Shipment log (Fig. 8), and document shipment on the Chain of Custody Record. A copy of the Chain of Custody records and Shipment Log will be retained. The original Chain of Custody Record and shipment log will remain with the sample(s).
- (d) The Materials Technician will include an aliquot chain of custody record (Fig. 9) with the original sample-id and aliquot sample-id recorded upon it for each aliquot to be generated at FDA. Solid food has one unique number. Liquid food and beverage have a different unique ID number.

At FDA, the composite solid food portion of the sample will be aliquoted into:

- (1) Food Metals Aliquot (prefix = 36, VID = FUM)
- (2) Food Pesticides Aliquot (prefix = 37, VID = FUP)
- (3) Solid Food Return Aliquot (prefix = 38, VID=FUR)

The liquid portion of the food sample will be aliquoted into:

- (1) Food Metals Aliquot (prefix = 36, VID = FUM)
- (2) Food Pesticides Aliquot (prefix = 37, VID = FUP)

- (e) A total of three aliquots will be generated from the solid food portion of the diet sample, and two aliquots will be generated from the liquid portion of the diet sample. Four barcoded copies of sample-id labels for each aliquot will be shipped with the Aliquot Chain of Custody records for use at FDA Labs.
- (f) All aliquots will be related to their original composite sample via the Aliquot Chain of Custody Record (fig.9) and through the NHEXAS Tracking System.
- (g) The Materials Technician will also complete the form letter to FDA Labs specifying analyses requested. A sample of this form letter is provided in figure 10. Note that the analyses requested **does not** change, but the sample-IDs will vary according to the shipment the letter accompanies.
- (h) Samples will be shipped in a protective container with sufficient blue-ice packs to maintain the samples at 4°C during express overnight mail to FDA Labs. Samples will be double-bagged to avoid cross-contamination and minimize spills during transport.
- (i) **Each sample shipped** will be accompanied by the following items secured in an

attached Ziploc Freezer Bag:

- (1) One Food Collection Data Sheet and Chain of Custody Record. (Fig. 3).
 - (2) Five Aliquot Chain of Custody Records (Fig. 9).
 - (3) Five unique barcode legible aliquot sample-IDs (4 copies of each ID for a total of 20 labels)
 - (4) One 24-Hour Food Diary Check sheet (Fig. 11).
 - (5) One copy of the appropriate 24-Hour Food Diary (Fig. 1).
- (j) **Each Shipment** will be accompanied by the following items secured in an attached Ziploc Freezer Bag:
- (1) One NHEXAS AZ Shipment Log (Fig. 8)
 - (2) One Form Letter to FDA Labs Specifying Analyses Requested (Fig. 10)
- (k) Up to four 50 mL solid food return aliquots will be returned by FDA to UA Labs for archive and further testing. These return aliquots will be kept frozen.

7.3 Analysis

7.3.1 Standards/Blanks

Field Blank standards, spiked samples and container lab blanks will be undergo the same handling and shipping as 'live blanks'. The samples collected from 10% of all residences sampled will be accompanied by a QA/QC blank or spike. Replicate or duplicate sampling of this duplicate sample methodology will not be accomplished due to financial and time burden on respondents.

7.3.2 Samples - N/A

7.4 Calculations - N/A

7.5 Quality Control

7.5.1 At the HH sampling site, the Team Leader supervises all work and forms completed. Team members work collectively and check each other's work for accuracy, precision and compliance with SOP procedure and policy.

7.5.2 Tolerance Limits

- (a) Temperature control is critical in food collection and sample integrity. Pesticides may volatilize with heat. The respondent will be instructed to keep all duplicate food and beverage samples refrigerated prior to collection by field staff.

- (b) Food will be transported from the field to the lab on blue ice at 4°C. The sample will be frozen prior to shipment and shipped on blue-ice.

7.5.3 Detection Limits

- (a) 100% of all data collected is field verified for accuracy.
- (b) Sample integrity must be maintained and is checked at each point of sample transfer.

7.5.4 Corrective Actions

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

7.5.5 Ten percent of all samples will be used for QA/QC purposes.

8.0 RECORDS

8.1 24-Hour Food Diary

8.1.1 This data record (Fig.1) will serve as the primary record of samples collected by the respondent in the field. The Team Leader assists the respondent with the thorough completion of this record. Enter data in the appropriate fields on the form and ensure correspondence with the food samples collected.

8.1.2 The completed original 24-Hour Food Diary will be archived with the HH packet after QA and QC checks. A photocopy of the diary will be maintained and archived by the Team Leader.

8.2 Food Diary Follow-up

8.2.1 This data record (Fig.2) will serve as the primary method for validating and extending the diary data provided by the respondent in the field. The Team Leader assists the respondent with the thorough completion of this record upon pick-up of the food samples.

8.2.2 The completed original Food Diary Follow-up will be archived with the HH packet after QA and QC checks.

Figure 1. 24-hour Food Diary (page 1 of 5)

OMB Clearance #: _____ - _____

Expires: _____

HHID# _____

NATIONAL HUMAN EXPOSURE ASSESSMENT SURVEY

24-HOUR FOOD DIARY

Respondent's First Name [ONLY] _____
Respondent's IRN # _____
Completed On _____ / _____ / _____ Mo Day Year

Public reporting burden for this collection of information is estimated to average ____ hours (or minutes) per response, and to require ____ hours recordkeeping. This includes the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing the burden, to Chief, Information Policy Branch, 2136, U.S. Environmental Protection Agency, 401 M St., S.W., Washington, D.C. 20460; and to the Office of Information and Regulatory Affairs, Office of Management and Budget, Washington, D.C. 20503.

OFFICE USE ONLY					
COMP	TYPE	TECH	SELF	GUARDIAN	OTHER
QC	INIT	MO	DAY	YR	
QA	INIT	MO	DAY	YR	
DE	INIT	MO	DAY	YR	
DP BATCH: _____					

Figure 1. 24-hour Food Diary (page 2 of 5)

NATIONAL HUMAN EXPOSURE ASSESSMENT SURVEY
24-HOUR FOOD DIARY QUESTIONNAIRE

What is the first name of the individual providing responses to these questions?

First name only IRN#

What is the relationship of this individual to the person for whom the responses are provided?

Check one:

☐ Self

☐ Guardian

☐ Other, specify _____

Is this a tech administered 24-Hour Food Diary QX?

Circle one. Y or N

TECH INITIALS _____ TECH ID _____

Comments:

Figure 1. 24-hour Food Diary (page 3 of 5)

HOW TO USE THE 24-HOUR FOOD DIARY

FOR PARTICIPANTS LESS THAN 10 YEARS OLD, A PARENT OR GUARDIAN SHOULD PROVIDE ASSISTANCE, AS NEEDED, IN COMPLETING THE FOOD DIARY.

INSTRUCTIONS

- (1) We want you to list all of the foods, beverages, drinking water, and non-prescription medicines or vitamins you or this child eat(s) or drink(s) from midnight to midnight.
- (2) Every time you or this child eat(s), write down the name of the meal (breakfast, lunch, dinner, snack).
- (3) Then write down on a separate line the (brand/generic) name of every food, beverage, or non-prescription medicine or vitamin that you or this child eat(s) or drink(s).
- (4) For food mixtures such as stews or potpies, please write down the major kinds of foods in the mixture. Use the lines immediately below the one on which the name of the mixture is entered. In food mixtures, the component ingredients can be identified, for example—the type of meat in a stew—beef, lamb, venison, etc.
- (5) For beverages (including water), write down how many cups or glasses that you or this child drink(s)). Estimate equivalent measures of water or other beverages taken from a fountain or large container. Don't forget your second and third cups of coffee or tea, or refills at a restaurant.

NHEXAS FOOD DIARY FOLLOW-UP - DAY 1 [NOTE: THE 24-HOUR FOOD DIARY WILL CONTAIN SIMILAR ENTRIES FOR EACH DAY ON WHICH DUPLICATE DIET SAMPLES ARE COLLECTED.]

[illegible]

Figure 2. Food Diary Follow-up (page 1 of 5)

HHID <div style="border: 1px solid black; width: 100px; height: 20px; margin: 0 auto;"></div>	F.S. <div style="border: 1px solid black; width: 20px; height: 20px; margin: 0 auto;"></div>	IRN # <div style="border: 1px solid black; width: 60px; height: 20px; margin: 0 auto;"></div>
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ADULT FOOD DIARY FOLLOW UP

National Human Exposure Assessment Survey

FIRST Name (ONLY): <div style="border: 1px solid black; width: 300px; height: 20px; margin: 0 auto;"></div>	Date of Completion: <div style="display: flex; justify-content: space-around; align-items: center;"><div style="border: 1px solid black; width: 30px; height: 20px; text-align: center;">MO</div><div style="border: 1px solid black; width: 30px; height: 20px; text-align: center;">DAY</div><div style="border: 1px solid black; width: 30px; height: 20px; text-align: center;">YR</div></div>
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INSTRUCTIONS

- Please use the **black, felt tip pen** provided in the notebook when you complete this questionnaire.
- Please follow the special guidelines typed in ***bold italics***. They tell you to either ***Continue*** to the question below, or to ***GO TO*** a given question.
- Please answer questions printed on a white background only. Shaded areas are for office use only.
- **Multiple Choice Questions:** Please fill in the appropriate bubble(s) (○) that appear to the left of each response.
For example: How old is the puppy? *Fill in ONE bubble.* ○ 1 week ● 2 weeks ○ 3 weeks

Please shade bubbles like this: ● and *not* like this: ✗ ✓
- **Open Ended Questions:** Please write your answer on the line(s) or in the box(es) provided. Please write your numbers in the boxes using a block style without touching the sides of the boxes.
For example:

0123456789

For example: How old is the puppy?

2

 week(s)
- **If You Make a Mistake:** For multiple choice, cross out the incorrect answer(s). For open-ended questions, cross out the incorrect value(s) and write the correct value(s) above or beside the boxes.
For example: How old is the puppy? *Fill in ONE bubble.* ✗ 1 week ● 2 weeks ○ 3 weeks
For example: How old is the puppy?

15
~~14~~

 week(s)
- If you wish to not answer a question, then please draw a line through it, *but not through the answer space*.
For example: ~~How old is the puppy?~~ week(s)

All data gathered in this questionnaire is kept strictly confidential. Thank you for your time and support!

Public reporting burden for this collection of information is estimated to average 5 minutes per response, and to require 0 hours of recordkeeping. This includes the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing the burden, to Chief, Information Policy Branch, 2136, U.S. Environmental Protection Agency, 401 M St., S.W., Washington, D.C. 20460; and to the Office of Information and Regulatory Affairs, Office of Management and Budget, Washington D.C. 20503. OMB Clearance #: 2080-0053 Expires: 07/31/98

Data Use Only:	0	1	2	3	4	5	6	7	8	9	A	B	C	D	E	F	G	H	I	J
	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>

25790



Study Participants: Please begin on page 3 (next page).

PAGE 2

Adult Food Follow Up

DESIGNATED PARTICIPANT

If the participant is LESS THAN 10 YEARS OLD, what is the first name of the individual who is providing the answers for the designated respondent?

FIRST Name (ONLY):

--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--

IRN #:

--	--

What is the relationship of this individual to the person for whom the responses are provided?
Fill in *ONE* bubble.

☐ 1. Self
☐ 2. Guardian
☐ 3. Other (Specify: _____)

OFFICE USE ONLY

☐ QC
☐ X's.R
☐ Y's.N
☐ Z's.M

☐ QC
☐ - 5.R
☐ - 8.N
☐ - 9.M

☐ 55.R
☐ 88.N
☐ 99.M

O Relat. Code

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Comments: _____

Chain of Custody initiated _____

Consigned to packet on []: ____/____/____

Box UA-G-4-2,0

OFFICE USE ONLY											
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Figure 2. Food Diary Follow-up (page 3 of 5)

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COMPLETE THIS ON THE SAME DAY THE FOOD SAMPLES ARE COLLECTED.		OFFICE USE ONLY																																					
1. Was breakfast eaten? <i>Fill in ONE bubble.</i> <input type="radio"/> 1. Yes <i>Continue below</i> <input type="radio"/> 2. No <i>GO TO Question # 4 (this page below)</i>		<div style="text-align: center;"> <input type="radio"/> R <input type="radio"/> N <input type="radio"/> M <input type="radio"/> 55 <input type="radio"/> 88 <input type="radio"/> 99 </div>																																					
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Figure 2. Food Diary Follow-up (page 4 of 5)

PAGE 4

Adult Food Follow Up

7. Was dinner eaten? *Fill in ONE bubble.*

☐ 1. Yes *Continue below*

☐ 2. No *GO TO Question # 10 (this page below)*

8. Where was your dinner prepared and eaten? *Read choices and shade bubble for PREPARED and EATEN for the location(s) that apply.*

	<u>PREPARED</u>	<u>EATEN</u>
a. Home	<input type="radio"/> P	<input type="radio"/> E
b. Restaurant or cafeteria	<input type="radio"/> P	<input type="radio"/> E
c. Work site	<input type="radio"/> P	<input type="radio"/> E
d. School or day care center	<input type="radio"/> P	<input type="radio"/> E
e. Other:	<input type="radio"/> P	<input type="radio"/> E

9. How often do you eat a dinner like the one you described in the diary? *Read choices and fill in ONE bubble.*

☐ 1. 6 to 7 times per week

☐ 2. 1 to 5 times per week

☐ 3. Less than once a week

10. Please think back. Were there any foods or beverages that you could not or did not collect for us? *Please list identity, source, and amount of each missing food.*

a. At breakfast:

☐ 1. Yes *Please LIST below*

☐ 2. No *GO TO Question # 10 b*

b. At lunch:

☐ 1. Yes *Please LIST below*

☐ 2. No *GO TO Question # 10 c*

c. At Dinner:

☐ 1. Yes *Please LIST below*

☐ 2. No *GO TO Question # 10 d (next page)*

OFFICE USE ONLY

R	N	M
<input type="radio"/> 55	<input type="radio"/> 88	<input type="radio"/> 99

O Loc

R	N	M
<input type="radio"/> 55	<input type="radio"/> 88	<input type="radio"/> 99
<input type="radio"/> 55	<input type="radio"/> 88	<input type="radio"/> 99
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<input type="radio"/> 55	<input type="radio"/> 88	<input type="radio"/> 99

R	N	M
<input type="radio"/> 55	<input type="radio"/> 88	<input type="radio"/> 99

B. Food 1

B. Food 2

B. Food 3

R	N	M
<input type="radio"/> 55	<input type="radio"/> 88	<input type="radio"/> 99

L. Food 1

L. Food 2

L. Food 3

R	N	M
<input type="radio"/> 55	<input type="radio"/> 88	<input type="radio"/> 99

D. Food 1

D. Food 2

D. Food 3

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Data Use Only:

<input type="radio"/> 0	<input type="radio"/> 1	<input type="radio"/> 2	<input type="radio"/> 3	<input type="radio"/> 4	<input type="radio"/> 5	<input type="radio"/> 6	<input type="radio"/> 7	<input type="radio"/> 8	<input type="radio"/> 9	<input type="radio"/> A	<input type="radio"/> B	<input type="radio"/> C	<input type="radio"/> D	<input type="radio"/> E	<input type="radio"/> F	<input type="radio"/> G	<input type="radio"/> H	<input type="radio"/> I	<input type="radio"/> J
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Figure 2. Food Diary Follow-up (page 5 of 5)

	PAGE 5																																												
<div style="display: flex; justify-content: space-between; margin-bottom: 10px;"> HHID: FS: IRN: </div> <p>10. (Continued) Please think back. Were there any foods or beverages that you could not or did not collect for us?</p> <p>d. For snacks -- include beverages such as coffee or tea</p> <p><input type="radio"/> 1. Yes Please LIST below</p> <p><input type="radio"/> 2. No GO TO Question # 11</p> <p>_____</p> <p>_____</p> <p>_____</p> <p>11. Did you, for any reason, eat <i>more or less</i> food than usual? Read choices and fill in ONE bubble.</p> <p><input type="radio"/> 1. More food than usual..... Continue below</p> <p><input type="radio"/> 2. Less food than usual Continue below</p> <p><input type="radio"/> 3. Same as usual GO TO Question # 13 (this page below)</p> <p>12. Because of... Read choices and fill in bubbles of ALL THAT APPLY.</p> <p><input type="radio"/> 1. Travel or vacation</p> <p><input type="radio"/> 2. Weight control diet</p> <p><input type="radio"/> 3. Illness or medical condition</p> <p><input type="radio"/> 4. Work or school schedule</p> <p><input type="radio"/> 5. Entertainment or social occasion</p> <p><input type="radio"/> 6. Because of the food collection study</p> <p><input type="radio"/> 7. Ease/quickness of preparation</p> <p><input type="radio"/> 8. Other (Specify: _____)</p> <p>13. Did you, for any reason, eat <i>different</i> foods than your usual diet? Fill in ONE bubble.</p> <p><input type="radio"/> 1. Yes Continue below</p> <p><input type="radio"/> 2. No STOP HERE</p> <p>14. Because of... Read choices and fill in bubbles of ALL THAT APPLY.</p> <p><input type="radio"/> 1. Travel or vacation</p> <p><input type="radio"/> 2. Weight control diet</p> <p><input type="radio"/> 3. Illness or medical condition</p> <p><input type="radio"/> 4. Work or school schedule</p> <p><input type="radio"/> 5. Entertainment or social occasion</p> <p><input type="radio"/> 6. Because of the food collection study</p> <p><input type="radio"/> 7. Ease/quickness of preparation</p> <p><input type="radio"/> 8. Other (Specify: _____)</p>	<p>OFFICE USE ONLY</p> <div style="margin-top: 10px;"> <p>R N M</p> <p><input type="radio"/> 55 <input type="radio"/> 88 <input type="radio"/> 99</p> <p>S. Food 1: </p> <p>S. Food 2: </p> <p>S. Food 3: </p> </div> <div style="margin-top: 10px;"> <p>R N M</p> <p><input type="radio"/> 55 <input type="radio"/> 88 <input type="radio"/> 99</p> <p> </p> </div> <div style="margin-top: 10px;"> <p>R N M</p> <p><input type="radio"/> 55 <input type="radio"/> 88 <input type="radio"/> 99</p> <p> </p> <p style="text-align: center;">O Rem.</p> <p style="text-align: center;"></p> </div> <div style="margin-top: 10px;"> <p>R N M</p> <p><input type="radio"/> 55 <input type="radio"/> 88 <input type="radio"/> 99</p> <p> </p> <p style="text-align: center;">O Rem.</p> <p style="text-align: center;"></p> </div>																																												
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Figure 3. Food Collection Data Sheet and Chain of Custody Record

Food Collection Data Sheet and Chain of Custody Record NHEXAS Arizona Food and Beverage Samples		
HHID# _____		
Participating Respondent's First Name : _____		
Sample Collection Date: ____/____/____ Participant's IRN _____ mo day yr		
FOOD SAMPLE ID# _____ OR BEVERAGE ID# _____		
SAMPLE COLLECTED BY FIELD TEAM ON ____/____/____ T LEADER = _____ mo day yr		
Sample Refrigerated by Respondent [Y] or [N] Samples compared to diet recorded in 24 Hour Food Diary [Y] or [N] Comments _____		
FOOD SAMPLE ALIQUOT IDENTIFICATION		
Metals Aliquot ID# _____		
Pesticides Aliquot ID # _____		
UA Return Aliquot ID # _____ or [] n/a		
Custody Record and 4 barcode labels of each aliquot type forwarded with original sample to FDA Lab ----> [] yes.		
Collection Comments: _____		Laboratory Comments _____
_____		_____
_____		_____
Custody Record		
<u>Custody of Sample</u> Name	Date mo./day/yr.	Operation Performed
_____	____/____/____	_____
_____	____/____/____	_____
_____	____/____/____	_____
_____	____/____/____	_____
_____	____/____/____	_____
_____	____/____/____	_____
_____	____/____/____	_____
_____	____/____/____	_____
_____	____/____/____	_____
_____	____/____/____	_____

Other Comments:

FORM = UA-F15.0-1.0

Figure 4. Subject instructions for the collection of food and beverages.

How to Collect Foods and Beverages

- Where we want you to collect food -

Please collect foods and beverages that you eat at home; this includes foods prepared somewhere else that you bring home to eat. Also collect foods and beverages that you prepare at home but might eat somewhere else.

- What we want you to collect -

Please prepare and collect a second identical portion of each food or beverage, that is, exactly the same kind and amount of food and beverage that you eat. Only include those items that you actually eat. Don't include orange peel, for example, unless you actually eat the orange peel. DO NOT include drinking water in your beverage container. We will also take a special sample directly from your water system later. Please eat the same foods you would have eaten if we were not asking you to collect a portion for us.

- When we want you to collect the food -

Please collect portions of all foods and beverages you eat or drink for the entire 24 hours of your assigned food collection day.

- How to collect the food -

- (a) At meal or snack time, prepare a second plate with the same type and same amounts of food you have added to your plate. Include all spices, sauces, butter, salt, ketchup, etc. Prepare a second cup, glass, or other container that holds the same amount of beverage that you will drink. Please use the same kind of plates, cups and glasses for the food you collect.
- (b) If you get more servings of food or beverage during your meal, add the same amount to the duplicate plate / beverage container.
- (c) If you are able, remove any inedible portions, like bones or pits, from foods on your second plate. (DON'T do this if you might get cut with a knife!).
- (d) At each meal and snack, write down the name and as many of the ingredients of the food and beverage that you ate on the diary we gave you.
- (e) Add the solid foods to the solid food containers where each meal is sealed in its own Ziploc freezer bag.
- (f) Add all beverages to the liquid food container. Frozen items that could melt, like ice cream or popsicles, should also be put in the liquid container. Re-seal the container.
- (g) **Remember to keep all samples in the refrigerator or cooler !**

Figure 5. Handling Diet Samples

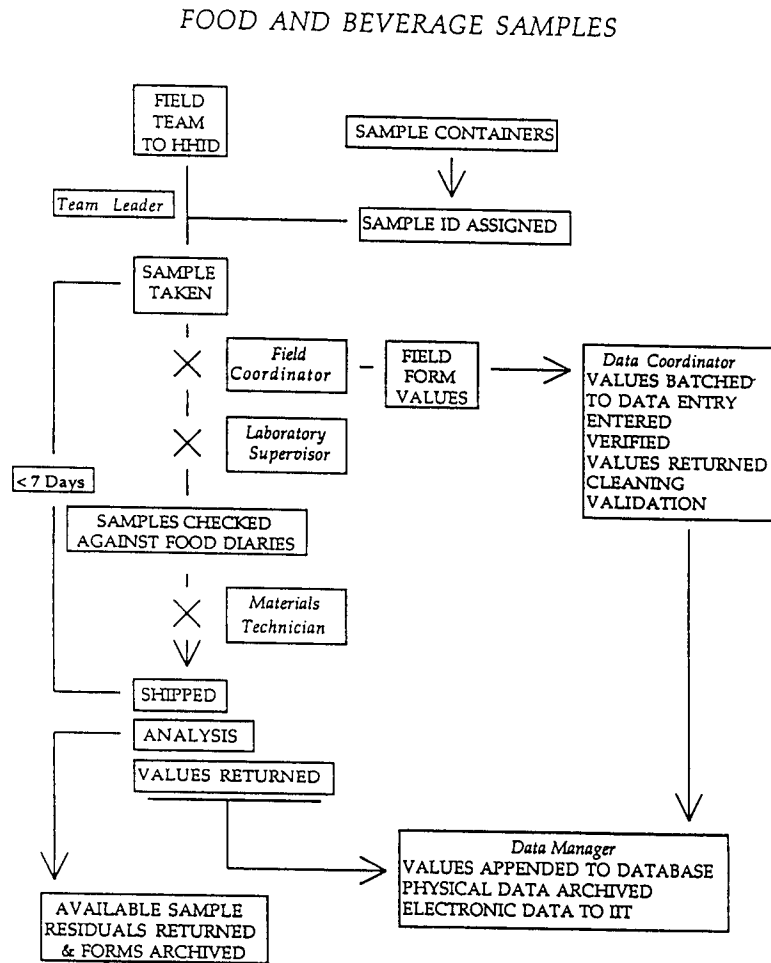


Figure 6. Troubleshooting Guide

No field notes or Troubleshooting guides are currently on record for UA-F-15.X. Additions will be appended and the SOP reviewed and updated in accordance with UA-G-1.X as appropriate.

Figure 7. Relative Timing of Sample Collection (page 1 of 3)

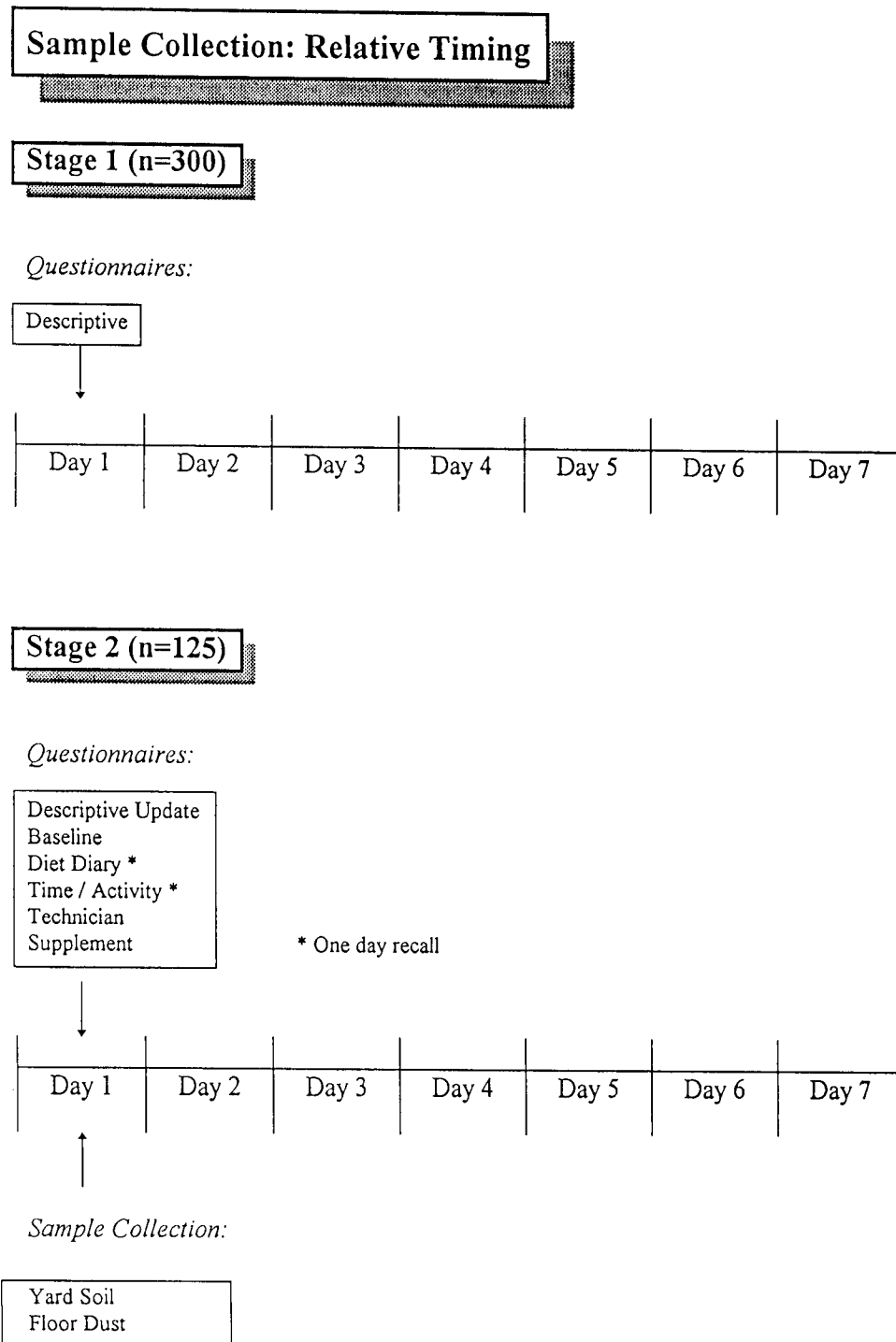
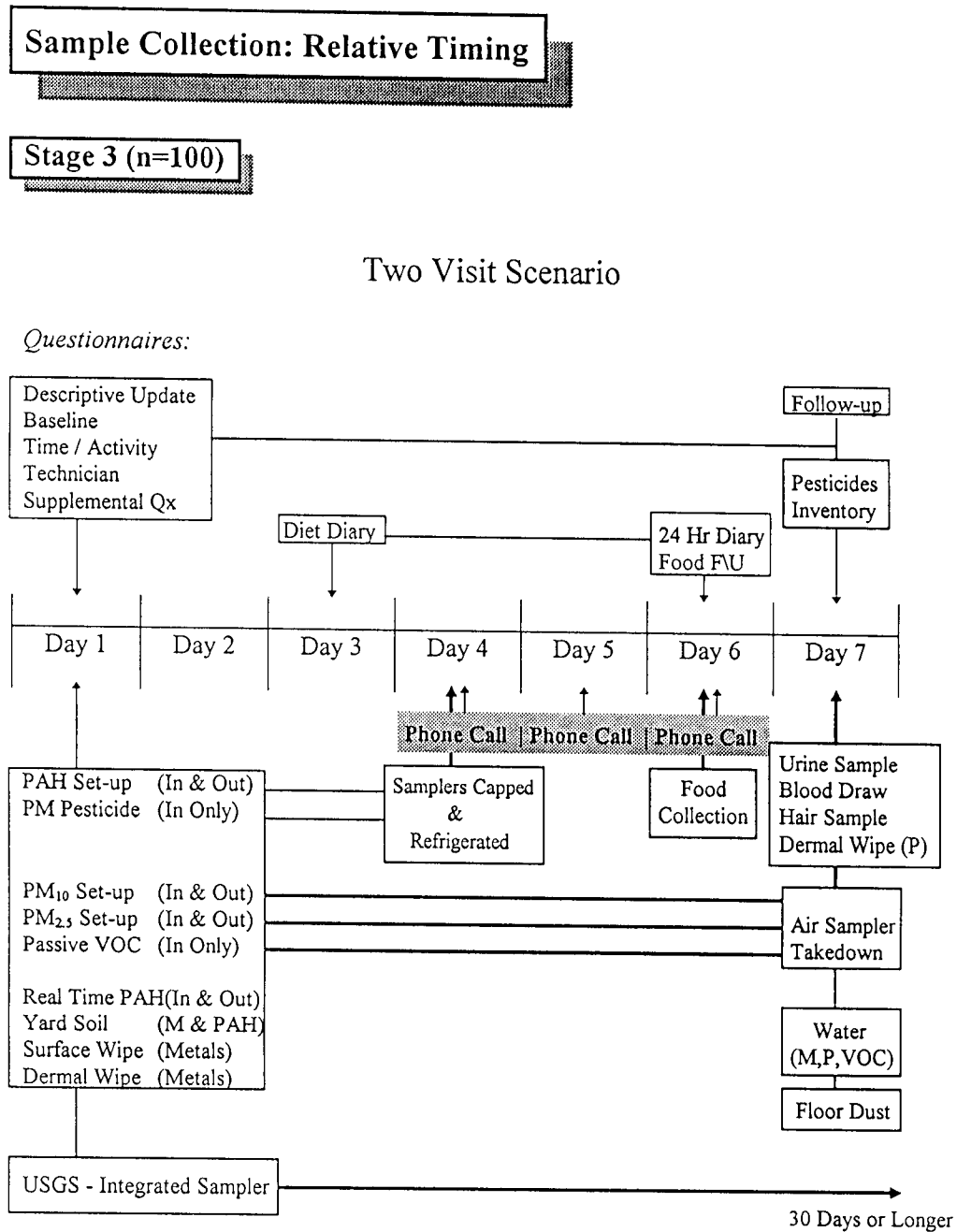
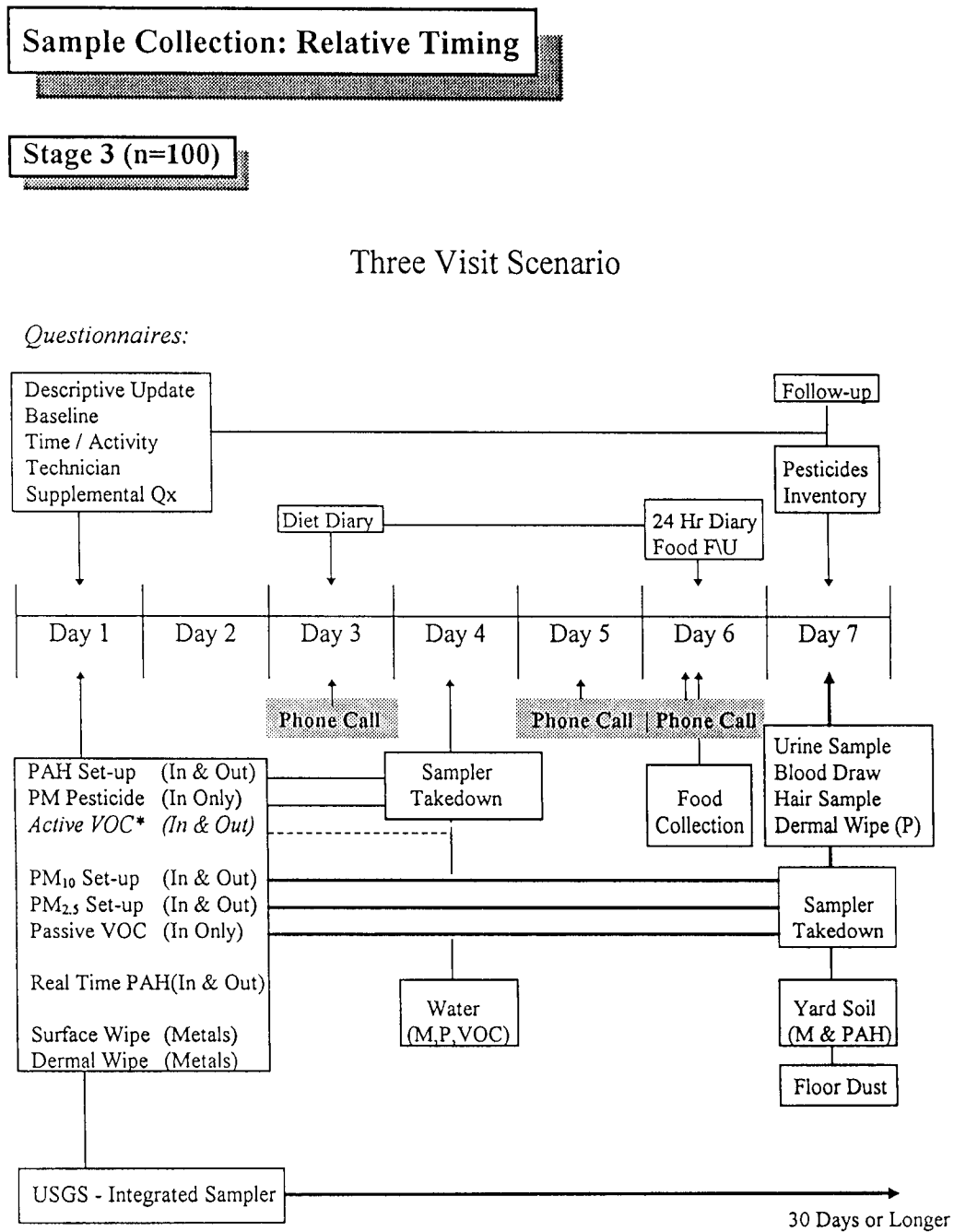


Figure 7. Relative Timing of Sample Collection (page 2 of 3)



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

Figure7. Relative Timing of Sample Collection (page 3 of 3)



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

FORM ID = UA-F2.0-1.0

[illegible]

FORM = UA-F-15.0-3.0

Figure 10. Form Letter to FDA Labs Specifying Analyses Requested. (SAMPLE)

Respiratory Sciences Center
A Division of the College of Medicine)

THE UNIVERSITY OF
ARIZONA
HEALTH SCIENCES CENTER

Tucson, Arizona 85724
FAX (602) 626-4884

15 March 1995

Food and Drug Administration
Kansas City District Office
11630 West 80 th Street
Lenexa, KS 66214

Attn: Kevin Cline
Re: Arizona NHEXAS Samples
AZ Contact: Mary Kay O'Rourke (602) 626-6835
Note: On March 20 our area code is supposed to change to 520

The following materials have been shipped overnight express mail and packed on blue ice:

Sample Number	Contents	Wt.	Analyses to be performed	Comments
1700002	Food	~490 g	Metals and Pesticides	
1700001	Beverage	2350 ml	" "	no alcohol
1700005	Food	~748 g	Metals and Pesticides	
1700006	Beverage	2350 ml	" "	no alcohol
1712852	Spike	750 ml		
1712121	Blank	750 ml		

SAMPLE

Please report the moisture contents as well as the analyte information.

Target Chemicals	CAS #	Target Chemical	CAS #
Primary Metals		Primary Pesticides	
Lead	7439-92-1	Chlorpyrifos	2921-88-2
Arsenic	7440-38-2	Diazinon	333-41-5
Cadmium	7440-43-9	Secondary Pesticides	
Nickel	7440-02-0	Malathion	121-75-5
Chromium	7440-47-3	Carbaryl	63-25-2
Secondary Metals			
Barium	7440-39-3		
Manganese	7439-96-5		
Selenium	7782-49-2		
Vanadium	7440-62-2		
Copper	7440-50-8		
Zinc	7440-66-6		

Please return up to 4 frozen aliquots in 50 ml plastic tubes (as per your protocol) along with the Blue ice and the styrofoam container. We understand that the return of aliquots is contingent on sufficient sample mass.

Thank you.

[illegible]

Appendix A. Foods mixed for Standards and Blanks (Page 1 of 3)

ITEM	TOTAL (grams)	CALCULATED GRAMS PER SAMPLE
*Broiled Ground Sirloin Lean Beef	630.0	12.95
*Skinless, White Boneless Chicken	644.0	13.23
Orleans Deveined Small Shrimp	120.0	2.47
Oysters	226.0	4.64
*Red Snapper with the Juice of 1 Lemon	590.0	12.12
Sardines in Mustard Sauce	106.0	2.18
Tuna	276.0	5.67
Ranch Style Beans (w/sliced Jalapenos)	425.0	8.73
Premium Dark Red Kidney Beans	432.0	8.88
Vegetarian Baked Beans	453.0	9.31
Bearitos Vegetarian Refried Black Beans	454.0	9.33
Rosarita Refried Beans	1150.0	22.63
Green Corn Tamales	374.5	7.70
Beef Tamales (2)	253.0	5.20
Pace Picante Sauce	680.0	13.97
Unpeeled fresh cooked potatoes	484.5	9.96
Cream style corn	454.0	9.33
Contadina Tomato Paste	200.0	4.11
Fresh Cooked Asparagus	531.2	10.92
Fresh Cooked Broccoli	165.0	3.39
*Fresh Celery	154.0	3.17
Fresh Cooked Cauliflower	166.0	3.41
*Fresh Carrots	508.7	10.45
*Fresh Green Pepper	131.5	2.70
*Fresh Mushrooms	227.0	4.67
Ortega Diced Chiles	113.0	2.32
*Romain Lettuce	349.0	7.17
*Fresh Unpeeled Cucumber	254.5	5.23
*Fresh Peeled Cucumber	191.5	3.94
Frozen Cooked Brussel Sprouts	453.0	9.31
Golden Hominy (canned)	265.0	5.45
Kale Greens	397.0	8.16
Mustard Greens	425.0	8.73
Bruces Cut Okra	397.0	8.16
Oriental Vegetables (cooked)	453.0	9.31
*Unpeeled Fresh apples	725.0	14.90
*Bananas Fresh	707.5	15.57
*Apples Fresh (peeled)	679.0	13.95

Appendix A. Foods mixed for Standards and Blanks (Page 2 of 3)

*Mixed fresh Apples, Oranges, & Bananas	452.0	9.29
Fruit Juice	185.0	3.80
Watermelon	1744.5	35.85
Strawberries	784.0	16.11
Apple Sauce	411.0	8.45
Bartlett Pears in Heavy Liquid	822.0	16.89
Dole Crushed Pineapple	227.0	4.67
Choice Fruit Cocktail	454.0	9.33
Del Monte Fruit Cup	110.0	2.26
Choice Yellow Cling Peaches in heavy syrup	425.0	8.73
*Fat Free Blueberry Muffins	390.0	8.02
*Whole Wheat Sunflower Seed Bread	160.0	3.29
*Bran and Raisin Muffin	75.5	1.55
*Flour Tortillas (4)	234.5	4.82
Crackers	78.0	1.60
Salt free Corn Chips	55.0	1.13
Pasta (cooked)	165.0	3.39
*Cooked Wild Rice	99.0	2.04
Eggs (6)	307.0	6.31
Cherry Vanilla Yogurt	250.0	5.14
Lemon Yogurt	250.0	5.14
Campbells Chunky Vegetable Soup	539.0	11.08
Cornbeef Hash	425.0	8.73
Don Miguel Frozen Mexican Dinner	326.0	6.70
Stouffers Spaghetti with Meat Sauce	326.0	6.70
*Entemanns Fat free Cinnamon Rolls	154.0	3.17
*McDonalds Egg McMuffin	127.5	2.62
*McDonalds Cheeseburger	108.5	2.23
*McDonalds French Fries	136.0	2.80
*McDonalds Breakfast Burrito	99.0	2.04
McDonalds Ketchup Packets	36.5	0.75
*Taco Bell Spicy Bean Burrito	209.0	4.30
*Taco Bell Regular Taco	161.0	3.31
*Pizza Hut Small Pan Pizza	287.0	5.90
Distilled water	907.0	18.64

Appendix A. Foods mixed for Standards and Blanks (Page 3 of 3)

Diet Coke	2268.0	46.61
Dr. Pepper	454.0	9.33
Milk	1814.0	37.28
Pepsi	1590.0	32.68
Alcohol Free Coors Cutters	340.0	6.99
Coffee (McDonalds)	680.0	13.97
Instant Coffee Powder	1.0	0.02
Cocoa	0.5	0.01
Carnation Sugar Free Cocoa (9 packages)	16.0	0.33
Chili Powder	1.0	0.02
Crushed Red Pepper	1.0	0.02
Cloves	5.0	0.10
Cinnamon	1.0	0.02
Oregano	1.0	0.02
Basil	1.0	0.02
Dill	1.0	0.02
Lee and Perrins	1.0	0.02
Salt	1.0	0.02
Pepper	1.75	0.05
Garlic Powder	0.25	0.01
Flour	75.0	1.54
Sugar	75.0	1.54
Raisins	75.0	1.54
Crunchy Blueberry Granola	134.5	2.76
Slim Fast Powder	33.5	0.69
Rhubarb Jelly	294.5	6.05
3- Bean Salad with Juice	425.0	8.73
Blueberry Pie (Safeway)	654.5	13.45
TOTAL:	35760.15	