

# National Human Exposure Assessment Survey (NHEXAS)

## *Region 5 Study*

## Quality Systems and Implementation Plan for Human Exposure Assessment

Research Triangle Institute  
Research Triangle Park, NC 27079

Cooperative Agreement CR 821902

**Field Operations Protocol**

**RTI/ACS-AP-209-003**

**Title:** Procedure for Collection, Storage, and Shipment of Drinking  
Water Samples for VOCs by EPA Method 524.2

**Source:** Research Triangle Institute

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  
OPERATIONS  
PROTOCOL**

**RESEARCH TRIANGLE INSTITUTE  
POST OFFICE BOX 12194  
RESEARCH TRIANGLE PARK, NC 27709-2194**

**RTVACS-AP-209-003**

**Page 1 of 10**

**TITLE:** PROCEDURE FOR COLLECTION, STORAGE, AND SHIPMENT OF  
DRINKING WATER SAMPLES FOR VOCs BY EPA METHOD 524.2

**SOURCE:** Research Triangle Institute  
Post Office Box 12194  
Analytical and Chemical Sciences  
Research Triangle Park, NC 27709-2194

**AUTHOR(s):**

Nent W. Thomas

Date: 4/8/96

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

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

**APPROVED BY:**

Principal Investigator:

E. Pelligrini

Date: 4/23/96

QA Officer:

D. J. Smith

Date: 4/11/96

<b><u>STATUS:</u></b>	IN PROGRESS:	<input type="checkbox"/>
	DRAFT:	<input type="checkbox"/>
	FINAL VERSION:	<input checked="" type="checkbox"/>

**REVISIONS:**

No.	Date	No.	Date
0	4/23/96	6	
1	†	7	
2		8	
3		9	
4		10	
5		11	

† Effective date of this version is the date of the last approval signature;  
revision 0 is the original version.

PROCEDURE FOR COLLECTION, STORAGE, AND SHIPMENT OF  
DRINKING WATER SAMPLES FOR VOCs BY EPA METHOD 524.2

TABLE OF CONTENTS

<u>Section</u>		<u>Page</u>
<a href="#">1.0</a>	<a href="#">Scope and Application</a> .....	3
<a href="#">2.0</a>	<a href="#">Summary of the Method</a> .....	3
<a href="#">3.0</a>	<a href="#">Sample Collection Materials</a> .....	3
<a href="#">4.0</a>	<a href="#">Preparation of Materials</a> .....	4
<a href="#">5.0</a>	<a href="#">Sample Collection</a> .....	4
<a href="#">6.0</a>	<a href="#">Sample Storage and Shipment</a> .....	5
<a href="#">7.0</a>	<a href="#">QC Procedures</a> .....	6

## 1.0 SCOPE AND APPLICATION

The procedures described in this protocol are designed only to guide the collection, storage, and shipping of drinking water samples for volatile organic chemicals (VOCs) for analysis by EPA Method 524.2 (version 4.0) during Phase I of the National Human Exposure Assessment Study (NHEXAS). Primary and secondary NHEXAS target analytes for this method are listed in Table 1. This protocol describes the sample containers, sample collection, preservation, storage, shipping and custody procedures.

## 2.0 SUMMARY OF THE METHOD

Sample containers will be prepared and shipped to the field site. The primary source of drinking water will be identified. Samples of the water will be collected in two separate vials. If the primary source is tap water, samples will be collected after running the water for three minutes. Ascorbic acid will be added to all samples to quench any possible residual chlorine. Hydrochloric acid will be added as a preservative. The samples will be chilled at 1E to 6EC from the time of collection until analysis. All samples will be shipped to the appropriate lab for analysis along with completed custody records for each sample.

### Method Reference

EPA Method 524.2, Revision 4.0, 8/92, U.S. EPA Methods for the Determination of Organic Compounds in Drinking Water, Office of Research and Development, Washington, D.C. (EPA/600/4-88/039).

## 3.0 SAMPLE COLLECTION MATERIALS

3.1 40 mL precleaned glass vials with Teflon lined silicone septa (Scientific Specialties Service, Inc., #376740, or equivalent) will be used.

3.2 Hydrochloric acid (36%-38% reagent grade).

3.3 Ascorbic Acid (Sigma ACS Reagent Grade 99%+ purity, or equivalent)

3.4 Cooler

3.5 Ice packs (Cole Palmer L-06346-70 or equivalent)

#### 4.0 PREPARATION OF MATERIALS

##### 4.1 Bottles

4.1.1 Precleaning will be by the supplier under EPA protocol B or (detergent wash, rinse with tap water, rinse with Type 1 water, oven dry, assemble in organic-free environment).

4.1.2 Alternatively, the vials will be washed according to ACS/SOP-150-001, "Standard Cleaning Procedure for Cleaning Glassware/Plasticware", and septa will be sonicated in methanol followed by vacuum oven drying.

##### 4.2 Hydrochloric Acid Preservative

4.2.1 Hydrochloric acid (1:1) will be prepared by adding 500 mL of concentrated hydrochloric acid to 400 mL of reagent grade water and diluting to a final volume of 1 L.

4.2.2 Two drops of 1:1 hydrochloric acid will be added to each 40 mL vial. The vial will be capped. The acid may be added to the vial at the laboratory prior to sample collection or at the field site during collection.

##### 4.3 Ascorbic Acid Quenching Agent

4.3.1 25 mg  $\pm$  5 mg of ascorbic acid will be weighed into clean 1 dram vials.

#### 5.0 SAMPLE COLLECTION

5.1 Have the participant identify the primary drinking water source.

5.2 If the water is from a tap, do not remove any aerators or disconnect any filters present at the tap and do not bypass any water softening systems.

5.3 If the water is from a tap, run the water at a moderate flow for at least three minutes.

5.4 If the water source is bottled water, do not run water prior to sample collection.

5.5 If the water source is a hand-pump from a well, pump for at least three minutes before sample collection.

5.6 Reduce the flow rate of the water to avoid introducing bubbles in the sample.

NOTE: Steps 5.1 through 5.5 can be eliminated if they have already been completed for another water sample at the home immediately prior to collection of these samples; the water flow should not be turned off between samples.

- 5.7 Add  $25 \pm 5$  mg of ascorbic acid to the vial immediately prior to filling.
- 5.8 Pour water slowly into the sample vial, do not pre-rinse, fill the vial to overflowing but do not flush out the ascorbic acid. Fill the vial so that a meniscus forms on top. Do not allow bubbles to form in the water during filling.
- 5.9 If hydrochloric acid is not already in the vial add two drops of 1:1 hydrochloric acid to reduce the pH to  $< 2$ .
- 5.10 Seal the vial with the septum Teflon face down, so that no air is trapped in the bottle.
- 5.11 Shake the vial vigorously for one minute.
- 5.12 Fill a second 40-mL vial, following steps 5.7 to 5.11.

## 6.0 SAMPLE STORAGE AND SHIPMENT

- 6.1 Immediately store the samples in the dark at 1E to 6EC.
- 6.2 The samples must be kept in the dark at 1E to 6EC at all times until analysis.
- 6.3 Before shipment, wrap electrical tape around the vial cap so that it does not loosen during shipment.
- 6.4 Ship the samples to the analytical laboratory within 7 days of collection.
- 6.5 In order to avoid breakage wrap the sample containers in bubble wrap, foam insert, or another protective cover so that it can not contact any other glass in the package.
- 6.6 Ship the samples in an insulated shipping container with sufficient cold packs so that the sample will remain cold for 24 hours.

NOTE: The samples must already be at 1E to 6EC when it is packed for shipment.

- 6.7 Enclose the sample custody records with the sample as it is shipped.
- 6.8 The samples should be shipped by overnight express method to the appropriate analysis lab.
- 6.9 Fax or mail a copy of the shipping summary sheet to EPA-Cincinnati.

## 7.0 QC PROCEDURES

### 7.1 Sample Code

- 7.1.1 A unique sample code will be assigned to each sample.
- 7.1.2 The sample container must have a label with a sample code identical to the code on the sample collection record. The sample label must be secured by winding clear tape over the label and completely around the bottle.

### 7.2 Custody

- 7.2.1 Complete the sample collection information in the sample collection record when the sample is collected. The information needed in the sample collection record is presented in Figure 1.
- 7.2.2 Enter the collector ID and date collected in the appropriate fields in the collection record.
- 7.2.3 Print the custody record prior to shipping the sample and enclose the original custody record with the sample as it is shipped.

### 7.3 Quality Control Samples

#### 7.3.1 Field Blanks

- 7.3.1.1 Field blanks are prepared to assess sample contamination from materials and methods.
- 7.3.1.2 Field blanks are prepared for a small percentage of the study homes, with the percentage defined in the QSIP.
- 7.3.1.3\* Field blanks are prepared in the field by adding contaminant-free water and  $25 \pm 5$  mg ascorbic acid to the sample collection container, with two drops of HCl (1:1).
- 7.3.1.4 Field blanks are then treated as a sample through storage and shipment.

#### 7.3.2 Collocated Sample Collection

- 7.3.2.1 Collocated samples are collected to assess collection and analysis precision.
- 7.3.2.2 Collocated samples are collected in a small percentage of homes, with the percentage defined in the QSIP.
- 7.3.2.3 Collocated samples are collected, stored and shipped following the same procedures as samples.

7.3.3 Field Controls

- 7.3.3.1 Field controls are prepared to assess recovery of target analytes through storage, shipment, and analysis.
- 7.3.3.2 Field controls are prepared for a small percentage of the study homes, to be defined in the QSIP.
- 7.3.3.3\* Field controls are prepared in the laboratory by adding a known amount of the target analytes to contaminant-free water in a sample collection container containing  $25 \pm 5$  mg ascorbic acid and two days of HCl (1:1).
- 7.3.3.4 Field controls are shipped to the field site, then taken to a participant's home and treated as a sample through storage and shipment to the analysis laboratory.



SAMPLE TYPE:	Drinking/VOC
SAMPLE CODE:	Same as label on container
PARTICIPANT ID:	Three digit participant i.d. number
COLLECTION DATE:	Date sample collected
COLLECTION TIME:	Time sample collected
COLLECTOR ID:	ID number of person that collected
COLLECTION LOCATION:	Default = kitchen tap; revise for other location/source
PRESERVATIVE ADDED (FIELD):	Default = 1:1 HCl
CHLORINE QUENCHER ADDED:	Default = Ascorbic acid
COMMENT CODE:	Default = 0; change to 1 or 2 if a comment is added below
COMMENT:	Add text for any comments associated with this particular sample.

Figure 1. Information to be included on the sample collection record.

TABLE 1. TARGET ANALYTES FOR NHEXAS PHASE I  
WATER COLLECTION METHOD 524.2

Primary	Secondary
Benzene	Methylchloroform
Chloroform	Methylene Chloride
Perchloroethylene	Styrene
Trichloroethylene	Toluene
	Xylenes ( <u>o</u> , <u>m</u> , <u>p</u> )
	<u>p</u> -Dichlorobenzene

## EXPLANATION OF REVISIONS

Revisions Made 4/96; Denoted by \*

Sections 7.3.1.3 and 7.3.3.3 revised to include ascorbic acid and HCl preservatives.