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Specifications and Guidance for Contaminant-Free Sample Containers

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SECTION I

INTRODUCTION

In August 1989, the Environmental Protection Agency's (EPA) Office of Emergency and Remedial Response (OERR) decentralized Superfund's Sample Container Repository program (OSWER Directive #9240.0-05). In conjunction with the decentralization of Superfund's bottle program, OERR issued specifications and guidance for preparing contaminant-free sample containers to assist the Regions in obtaining appropriate sample containers from commercially available suppliers.

The December 1992 version of "Specifications and Guidance for Contaminant-Free Sample Containers" revises the specifications and provides a single source of standardized specifications and guidance on appropriate cleaning procedures for preparing contaminant-free sample containers that meet all Contract Laboratory Program (CLP) detection/quantitation limits, including those for low concentration analyses.[1] Although the specifications and guidance procedures contained in this document are based on CLP low concentration requirements, they also are suitable for use in other analytical programs.

Specifications and guidance for preparing contaminant-free sample containers are provided in the sections that follow and are intended to describe one approach for obtaining cleaned, contaminant-free sample containers for use by groups performing sample collection activities under Superfund and other hazardous waste programs. Although other cleaning procedures may be used, sample containers must meet the criteria specified in Section II. In certain instances, the user of the sample containers may require exact adherence to the cleaning procedures and/or quality control analysis described in this document. In other instances, the user may require additional or different cleaning procedures and/or quality control analysis of the sample containers. The specific needs of the bottle user will determine the requirements for the cleaning and quality control analysis of the sample containers as long as the minimum criteria are met. It is the responsibility of the bottle user to define the sample container preparation, cleaning, and quality control requirements.

The document has been extensively reviewed and revised since the August 1989 iteration, and important enhancements have been incorporated, including:

- Removing references to the color of the closures;
- Allowing the use of polypropylene closures as an alternative to phenolic closures;
- Referencing CLP Low Concentration Organics and Inorganics Statements of Work for the analysis of calibration verification solutions and blanks;
- Including cleaning and quality control procedures for fluoride and nitrate/nitrite;
- Removing the hexane rinse from the cleaning procedure for container types A, E, F, G, H, J, and K (semivolatile organics, pesticides, metals, cyanide, and fluoride in soils and water);

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[1] Because this document does not address the procurement of contaminant-free sample containers, the title was changed from "Specifications and Guidance for Obtaining Contaminant-Free Sample Containers" to "Specifications and Guidance for Contaminant-Free Sample Containers."

- Adding the recommendation that the bottle vendor establish and submit a Quality Assurance Plan (QAP);

- Changing the QA/QC documentation requirements so that copies of the raw data from the analyses of the QC containers are available upon request and not automatically sent to the bottle purchaser;

- Changing the permanent lot number assignment to a nine-digit number from an eight-digit number, where the extra digit represents the analysis parameter;

- Adding Chemical Abstract Services (CAS) registry number for the inorganic analytes in Table 1; and

- Recommending an annual demonstration of the bottle vendor's ability to meet detection limits and establish reproducibility of the cleaning techniques.

OERR and the EPA Regions decided to use the most stringent CLP requirements available to set the specifications for obtaining contaminant-free sample containers. As a result, the CLP Inorganics and Organics Low Concentration Statement of Work (SOW) requirements were selected as the basis for these specifications. Major factors in this decision included the desire to have a set of bottle cleaning specifications that met or exceeded all analytical requirements and the related need to avoid potential misuse of cleaned bottles (e.g., using a container cleaned by a multi-concentration procedure for a low concentration sample). OERR will reevaluate this decision if the low concentration requirements are deemed to be too stringent.

Most environmental sampling and analytical applications offer numerous opportunities for sample contamination. For this reason, contamination is a common source of error in environmental measurements. The sample container itself represents one such source of sample contamination. Hence, it is vital that sample containers used within the Superfund program meet strict specifications established to minimize contamination which could affect subsequent analytical determinations. Superfund sampling and analysis activities require all component materials (caps, liners, septa, packaging materials, etc.) provided by the bottle preparer to meet the criteria limits of the bottle specifications listed within Section II.

Section III provides guidance on cleaning procedures for preparing contaminant-free sample containers that meet the specifications contained in Section II. The procedures provided in this section are intended to provide sample containers that meet all current CLP Low Concentration Inorganics and Organics detection/quantitation levels.

In selecting cleaning procedures for sample containers, it is important to consider all of the parameters of interest. Although a given cleaning procedure may be effective for one parameter or type of analysis, it may be ineffective for another. When multiple determinations are performed on a single sample or on a subsample from a single container, a cleaning procedure may actually be a source of contamination for some analytes while minimizing contamination in others. It should be the responsibility of the bottle supplier to verify that the cleaning procedures actually used satisfy the quality control requirements set forth in Section IV.

Two aspects of quality assurance (i.e., quality control and quality assessment) must be applied to sample containers as well as to the analytical measurements. Quality control includes the application of good laboratory practices and standard operating procedures especially designed for the cleaning of sample containers. The cleaning operation should be based on protocols especially designed for specific contaminant problems. Strict adherence to these cleaning protocols is imperative. Quality assessment of the cleaning process depends largely on monitoring for adherence to the respective protocols. Because of their critical role in the quality assessment of the cleaning operation, protocols must be carefully designed and followed. Guidance is provided in Section IV on design and implementation of quality assurance and quality control protocols.

SECTION II

SAMPLE CONTAINER AND COMPONENT MATERIAL SPECIFICATIONS

This Section identifies sample containers commonly used in the Superfund program and provides specifications for contaminant-free sample containers for each bottle type.

A. CONTAINER MATERIAL

A variety of factors affect the choice of containers and cap material. These include resistance to breakage, size, weight, interferences with analytes of interest, cost, and availability.

Container types A through L (Figure 1, pages 6-7) are designated as the type of sample containers that have been used successfully in the past. Kimax or Pyrex brand borosilicate glass is inert to most materials and is recommended where glass containers are used (i.e., pesticides and other organics). Conventional polyethylene is recommended when plastic is acceptable because of its lower cost and lower adsorption of metal ions. The specific sampling situation will determine the use of plastic or glass.

While the sample containers shown in Figure 1 are utilized primarily for Superfund sampling activities, they also may be used for sampling activities under other programs, such as the Resource Conservation and Recovery Act (RCRA).

B. MAXIMUM CONTAMINANT LEVEL SPECIFICATIONS FOR SAMPLE CONTAINERS

The CLP, through a series of technical caucuses, has established inorganic Contract Required Detection Limits (CRDL) and organic Contract Required Quantitation Limits (CRQL) which represent the minimum quantities needed to support the hazardous substance identification and monitoring requirements necessary for remedial and other actions at hazardous waste sites.

For inorganic sample containers, the CRDLs listed in Table 1, page 8, are the specifications for maximum trace metal contamination. Concentration at or above these limits on any parameter should preclude these containers from use in collecting inorganic samples.

The CRQL specifications for organic sample containers are listed in Table 2, pages 9-13. When the CRQL in Table 2 is multiplied by the appropriate factor listed below, the resulting value then represents the maximum concentration allowed for particular sample containers based on organic CLP sample sizes for routine analyses.

Container type	Multiple of CRQL
A	1.0
B	0.5
D	10.0
E	8.0
F	4.0
G	2.0
H	0.5
J	0.5
K	2.0

The philosophy used for determining the maximum permissible amount of contamination in a sample container was to consider the number of aliquots of sample that are available in the container and assume that the contamination present would be uniformly distributed in all of the aliquots. This assumption, and the assumption that there should be no more than one-half the CRQL contributed by the container, resulted in the establishment of contamination limits by container type. For example, the volume of container type D is sufficient to allow 20 volatile determinations. Therefore, if 10 times the CRQL of contaminant is present in the cleaned bottle, each aliquot tested will contain one-half of the CRQL of contaminant due to the contribution from the bottle.

C. GROSS CONTAMINATION

Gross contamination is defined as greater than two hundred times the acceptable concentration values in Tables 1 or 2 (multiplied by the appropriate factor), unless the cleaning procedure is successful in reducing the amount of contamination to within specifications. If this is not achieved, the grossly contaminated materials should be discarded and replaced to prevent cross contamination with other batches of containers. The bottle preparer should inspect all materials to ensure conformance with the required specifications.

FIGURE 1

SAMPLE CONTAINER SPECIFICATIONS

Container Type	Specifications
A	Container: 80-oz amber glass, ring handle bottle/jug, 38-mm neck finish. Closure: polypropylene or phenolic cap, 38-430 size; 0.015-in Teflon liner. Total Weight: 2.45 lbs.
B	Container: 40-mL glass vial, 24-mm neck finish. Closure: polypropylene or phenolic, open-top, screw cap, 15-cm opening, 24-400 size. Septum: 24-mm disc of 0.005-in Teflon bonded to 0.120-in silicon for total thickness of 0.125-in. Total Weight: 0.72 oz.
C	Container: 1-L high-density polyethylene, cylinder-round bottle, 28-mm neck finish. Closure: polyethylene cap, ribbed, 28-410 size; F217 polyethylene liner. Total Weight: 1.89 oz.
D	Container: 120-mL wide mouth, glass vial, 48-mm neck finish. Closure: polypropylene cap, 48-400 size; 0.015-in Teflon liner. Total Weight: 4.41 oz.
E	Container: 16-oz tall, wide mouth, straight-sided, flint glass jar, 63-mm neck finish. Closure: polypropylene or phenolic cap, 63-400 size; 0.015-in Teflon liner. Total Weight: 9.95 oz.
F	Container: 8-oz short, wide mouth, straight-sided, flint glass jar, 70-mm neck finish. Closure: polypropylene or phenolic cap, 70-400 size; 0.015-in Teflon liner. Total Weight: 7.55 oz.

FIGURE 1

SAMPLE CONTAINER
SPECIFICATIONS
(Continued)

Container Type	Specifications
G	Container: 4-oz tall, wide mouth, straight-sided, flint glass jar, 48-mm neck finish. Closure: polypropylene or phenolic cap, 48-400 size; 0.015-in Teflon liner. Total Weight: 4.70 oz.
H	Container: 1-L amber, Boston round, glass bottle, 33-mm pour-out neck finish. Closure: polypropylene or phenolic cap, 33-430 size; 0.015-in Teflon liner. Total Weight: 1.11 lbs.
J	Container: 32-oz tall, wide mouth, straight-sided, flint glass jar, 89-mm neck finish. Closure: polypropylene or phenolic cap, 89-400 size; 0.015-in Teflon liner. Total Weight: 1.06 lbs.
K	Container: 4-L amber glass, ring handle bottle/jug, 38-mm neck finish. Closure: polypropylene or phenolic cap, 38-430 size; 0.015-in Teflon liner. Total Weight: 2.88 lbs.
L	Container: 500-mL high-density polyethylene, cylinder-round bottle, 28-mm neck finish. Closure: polypropylene cap, ribbed, 28-410 size; F217 polyethylene liner. Total Weight: 1.20 oz.

TABLE 1

INORGANIC ANALYTE
SPECIFICATIONS

	Analyte	CAS Number	CRDL[1] (µg/L)
1.	Aluminum	7429-90-5	100
2.	Antimony	7440-36-0	5
3.	Arsenic	7440-38-2	2
4.	Barium	7440-39-3	20
5.	Beryllium	7440-41-7	1
6.	Cadmium	7440-43-9	1
7.	Calcium	7440-70-2	500
8.	Chromium	7440-47-3	10
9.	Cobalt	7440-48-4	10
10.	Copper	7440-50-8	10
11.	Iron	7439-89-6	500
12.	Lead	7439-92-1	2
13.	Magnesium	7439-95-4	500
14.	Manganese	7439-96-5	10
15.	Mercury	7439-97-6	0.2
16.	Nickel	7440-02-0	20
17.	Potassium	7440-09-7	750
18.	Selenium	7782-49-2	3
19.	Silver	7440-22-4	10
20.	Sodium	7440-23-5	500
21.	Thallium	7440-28-0	10
22.	Vanadium	7440-62-2	10
23.	Zinc	7440-66-6	20
24.	Cyanide	57-12-5	10
25.	Fluoride	16984-48-8	200
26.	Nitrate/Nitrite	1-005	100

[1] CRDLs are based on the CLP Inorganics Low Concentration SOW

TABLE 2

ORGANIC COMPOUND SPECIFICATIONS

	Volatiles	CAS Number	CRQL[1] (µg/L)
1.	Chloromethane	74-87-3	1
2.	Bromomethane	74-83-9	1
3.	Vinyl Chloride	75-01-4	1
4.	Chloroethane	75-00-3	1
5.	Methylene Chloride	75-09-2	2
6.	Acetone	67-64-1	5
7.	Carbon Disulfide	75-15-0	1
8.	1,1-Dichloroethene	75-35-4	1
9.	1,1-Dichloroethane	75-34-3	1
10.	cis-1,2-Dichloroethene	156-59-4	1
11.	trans-1,2-Dichloroethene	156-60-5	1
12.	Chloroform	67-66-3	1
13.	1,2-Dichloroethane	107-06-2	1
14.	2-Butanone	78-93-3	5
15.	Bromochloromethane	74-97-5	1
16.	1,1,1-Trichloroethane	71-55-6	1
17.	Carbon Tetrachloride	56-23-5	1
18.	Bromodichloromethane	75-27-4	1
19.	1,2-Dichloropropane	78-87-5	1
20.	cis-1,3-Dichloropropene	10061-01-5	1
21.	Trichloroethene	79-01-6	1
22.	Dibromochloromethane	124-48-1	1
23.	1,1,2-Trichloroethane	79-00-5	1
24.	Benzene	71-43-2	1
25.	trans-1,3-Dichloropropene	10061-02-6	1
26.	Bromoform	75-25-2	1
27.	4-Methyl-2-pentanone	108-10-1	5
28.	2-Hexanone	591-78-6	5
29.	Tetrachloroethene	127-18-4	1
30.	1,1,2,2-Tetrachloroethane	79-34-5	1

[1] CRQLs are based on the CLP Organics Low Concentration SOW

TABLE 2 (cont.)

ORGANIC COMPOUND SPECIFICATIONS

	Volatiles	CAS Number	CRQL[1] (μg/L)
31.	1,2-Dibromoethane	106-93-4	1
32.	Toluene	108-88-3	1
33.	Chlorobenzene	108-90-7	1
34.	Ethylbenzene	100-41-4	1
35.	Styrene	100-42-5	1
36.	Xylenes (total)	1330-20-7	1
37.	1,3-Dichlorobenzene	541-73-1	1
38.	1,4-Dichlorobenzene	106-46-7	1
39.	1,2-Dichlorobenzene	95-50-1	1
40.	1,2-Dibromo-3-chloropropane	96-12-8	1

[1] CRQLs are based on the CLP Organics Low Concentration SOW

TABLE 2 (cont.)

ORGANIC COMPOUND
SPECIFICATIONS

	Semivolatiles	CAS Number	CRQL[1] (μg/L)
1.	Phenol	108-95-2	5
2.	bis-(2-Chlorethyl)ether	111-44-4	5
3.	2-Chlorophenol	95-57-8	5
4.	2-Methylphenol	95-48-7	5
5.	2,2'-oxybis-(1-Chloropropane)	108-60-1	5
6.	4-Methylphenol	106-44-5	5
7.	N-Nitroso-di-n-dipropylamine	621-64-7	5
8.	Hexachloroethane	67-72-1	5
9.	Nitrobenzene	98-95-3	5
10.	Isophorone	78-59-1	5
11.	2-Nitrophenol	88-75-5	5
12.	2,4-Dimethylphenol	105-67-9	5
13.	bis-(2-Chloroethoxy)methane	111-91-1	5
14.	2,4-Dichlorophenol	120-83-2	5
15.	1,2,4-Trichlorobenzene	120-82-1	5
16.	Naphthalene	91-20-3	5
17.	4-Chloroaniline	106-47-8	5
18	Hexachlorobutadiene	87-68-3	5
19.	4-Chloro-3-methylphenol	59-50-7	5
20.	2-Methylnaphthalene	91-57-6	5
21.	Hexachlorocyclopentadiene	77-47-4	5
22.	2,4,6-Trichlorophenol	88-06-2	5
23.	2,4,5-Trichlorophenol	95-95-4	20
24.	2-Chloronaphthalene	91-58-7	5
25.	2-Nitroaniline	88-74-4	20
26.	Dimethylphthalate	131-11-3	5
27.	Acenaphthylene	208-96-8	5
28.	2,6-Dinitrotoluene	606-20-2	5
29.	3-Nitroaniline	99-09-2	20
30.	Acenaphthene	83-32-9	5

[1] CRQLs are based on the CLP Organics Low Concentration SOW

TABLE 2 (cont.)

ORGANIC COMPOUND
SPECIFICATIONS

	Semivolatiles	CAS Number	CRQL[1] (μg/L)
31.	2,4-Dinitrophenol	51-28-5	20
32.	4-Nitrophenol	100-02-7	20
33.	Dibenzofuran	132-64-9	5
34.	2,4-Dinitrotoluene	121-14-2	5
35.	Diethylphthalate	84-66-2	5
36.	4-Chlorophenyl-phenylether	7005-72-3	5
37.	Fluorene	86-73-7	5
38.	4-Nitroaniline	100-01-6	20
39.	4,6-Dinitro-2-methylphenol	534-52-1	20
40.	N-Nitrosodiphenylamine	86-30-6	5
41.	4-Bromophenyl-phenylether	101-55-3	5
42.	Hexachlorobenzene	118-74-1	5
43.	Pentachlorophenol	87-86-5	20
44.	Phenanthrene	85-01-8	5
45.	Anthracene	120-12-7	5
46.	Di-n-butylphthalate	84-74-2	5
47.	Fluoranthene	206-44-0	5
48	Pyrene	129-00-0	5
49.	Butylbenzylphthalate	85-68-7	5
50.	3,3'-Dichlorobenzidine	91-94-1	5
51.	Benz[a]anthracene	56-55-3	5
52.	Chyrsene	218-01-9	5
53.	bis-(2-Ethylhexyl)phthalate	117-81-7	5
54.	Di-n-octylphthalate	117-84-0	5
55.	Benzo[b]fluoranthene	205-99-2	5
56.	Benzo[k]fluoranthene	207-08-9	5
57.	Benzo[a]pyrene	50-32-8	5
58.	Indeno(1,2,3-cd)pyrene	193-39-5	5
59.	Dibenz[a,h]anthracene	53-70-3	5
60.	Benzo[g,h,i]perylene	191-24-2	5

[1] CRQLs are based on the CLP Organics Low Concentration SOW

TABLE 2 (cont.)

ORGANIC COMPOUND SPECIFICATIONS

	Pesticides/PCBs	CAS Number	CRQL[1] (μg/L)
1.	alpha-BHC	319-84-6	0.01
2.	beta-BHC	319-85-7	0.01
3.	delta-BHC	319-86-8	0.01
4.	gamma-BHC (Lindane)	58-89-9	0.01
5.	Heptachlor	76-44-8	0.01
6.	Aldrin	309-00-2	0.01
7.	Heptachlor epoxide	1024-57-3	0.01
8.	Endosulfan I	959-98-8	0.01
9.	Dieldrin	60-57-1	0.02
10.	4,4'-DDE	72-55-9	0.02
11.	Endrin	72-20-8	0.02
12.	Endosulfan II	33213-65-9	0.02
13.	4,4'-DDD	72-54-8	0.02
14.	Endosulfan sulfate	1031-07-8	0.02
15.	4,4'-DDT	50-29-3	0.02
16.	Methoxychlor	72-43-5	0.10
17.	Endrin ketone	53494-70-5	0.02
18	Endrin aldehyde	7421-36-3	0.02
19.	alpha-Chlordane	5103-71-9	0.01
20.	gamma-Chlordane	5103-74-2	0.01
21.	Toxaphene	8001-35-2	1.0
22.	Aroclor-1016	12674-11-2	0.20
23.	Aroclor-1221	11104-28-2	0.20
24.	Aroclor-1232	11141-16-5	0.40
25.	Aroclor-1242	53469-21-9	0.20
26.	Aroclor-1248	12672-29-6	0.20
27.	Aroclor-1254	11097-69-1	0.20
28.	Aroclor-1260	11096-82-5	0.20

[1] CRQLs are based on the CLP Organics Low Concentration SOW

SECTION III

SAMPLE CONTAINER PREPARATION AND CLEANING PROCEDURES

This Section is provided as guidance for the preparation of sample containers that meet the contaminant-free specifications contained in Section II. There are various procedures for cleaning sample containers depending upon the analyses to be performed on the sample. The following cleaning procedures are modeled after those specified for the Superfund Sample Container Repository program. Other suitable cleaning procedures exist and may be used as long as the sample containers meet the criteria established in Section II. In some instances, the specific needs of the bottle user may dictate exact adherence to the sample container preparation and cleaning procedures that follow; while in other instances, modifications may be required. It is the responsibility of the bottle user to define the sample container preparation, cleaning, and quality control requirements.

A. Cleaning Procedure for Container Types: A, E, F, G, H, J, and K

1. Sample Type: Semivolatile Organics, Pesticides, Metals, Cyanide, and Fluoride in Soils and Water.

   a. Wash glass bottles, Teflon liners, and caps with hot tap water using laboratory grade nonphosphate detergent.

   b. Rinse three times with copious amounts of tap water to remove detergent.

   c. Rinse with 1:1 nitric acid (reagent grade HNO_3, diluted with ASTM Type I deionized water).

   d. Rinse three times with ASTM Type I organic free water.

   e. Oven dry bottles, liners, and caps at 105-125°C for one hour.

   f. Allow bottles, liners, and caps to cool to room temperature in an enclosed contaminant-free environment.

   g. Rinse bottles with pesticide grade methylene chloride (or other suitable solvents specified by the bottle user) using 20 mL for ½-gallon containers; 10 mL for 32-oz and 16-oz containers; and 5 mL for 8-oz and 4-oz containers.

   h. Oven dry bottles, liners, and caps at 105-125°C for one hour.

   i. Allow bottles, liners, and caps to cool to room temperature in an enclosed contaminant-free environment.

   j. Place liners in lids and cap containers.

   k. Label each container with the lot number and pack in a case.

   l. Label exterior of each case with the lot number.

   m. Store in a contaminant-free area.

2. Sample Type: Nitrate/Nitrite in Soils and Water.

   a. Substitute reagent grade sulfuric acid (H_2SO_4) for nitric acid in step A.1.c.

   b. Follow all other steps in the cleaning procedure described in part A.1 above.

B. Cleaning Procedure for Container Types: B, D

1. Sample Type: Purgeable (Volatile) Organics in Soils and Water.

   a. Wash glass vials, Teflon-backed septa, Teflon liners, and caps in hot water using laboratory grade nonphosphate detergent.

   b. Rinse three times with copious amounts of tap water to remove detergent.

   c. Rinse three times with ASTM Type I organic-free water.

   d. Oven dry vials, caps, septa, and liners at 105-125°C for one hour.

   e. Allow vials, caps, septa, and liners to cool to room temperature in an enclosed contaminant-free environment.

   f. Seal 40-mL vials with septa (Teflon side down) and cap.

   g. Place liners in lids and cap 120-mL vials.

   h. Label each vial with the lot number and pack in a case.

   i. Label exterior of each case with the lot number.

   j. Store in a contaminant-free area.

C. Cleaning Procedure for Container Types: C, L

1. Sample Type: Metals, Cyanide, and Fluoride in Soils and Water.

   a. Wash polyethylene bottles and caps in hot tap water using laboratory-grade nonphosphate detergent.

   b. Rinse three times with copious amounts of tap water to remove detergent.

   c. Rinse with 1:1 nitric acid (reagent grade HNO_3, diluted with ASTM Type I deionized water).

   d. Rinse three times with ASTM Type I deionized water.

   e. Invert and air dry in a contaminant-free environment.

   f. Cap bottles.

   g. Label each container with the lot number and pack in a case.

h. Label exterior of each case with the lot number.

i. Store in a contaminant-free area.

2. Sample Type: Nitrate/Nitrite in Soils and Water.

a. Substitute reagent grade sulfuric acid (H_2SO_4) for nitric acid in step C.1.c.

b. Follow all other steps in the cleaning procedure described in part C.1 above.

SECTION IV

SAMPLE CONTAINER QUALITY ASSURANCE AND QUALITY CONTROL REQUIREMENTS

A. Quality Assurance

The objectives of this Section are to: (1) present procedures for evaluating quality assurance (QA) information to ensure that specifications identified in Section II have been met; and (2) discuss techniques for the quality control (QC) analysis of sample containers to be used in conjunction with the cleaning procedures contained in Section III.

The bottle vendor should establish a Quality Assurance Plan (QAP) with the objective of providing sound analytical chemical measurements, production procedures, and tracking systems. The QAP should incorporate procedures for the inspection of incoming raw materials; preparation, cleaning, and labeling of container lots; quality control analyses of cleaned container lots; document control, including all documentation required for analysis, packing, shipping, and tracking of container lots; any necessary corrective actions; and any quality assessment measures implemented by management to ensure acceptable performance. The QAP should be available and provided to the bottle purchaser upon request.

Major QA/QC activities should include the inspection of all incoming materials, QC analysis of cleaned lots of containers, and monitoring of the container storage area. Complete documentation of all QC inspection results (acknowledging acceptance or rejection) should be kept as part of the permanent bottle preparation files. QA/QC records (e.g., preparation/QC logs, analytical data, data tapes, storage log) also should be stored in a central location within the facility.

Documentation indicating that the container lot has passed all QA/QC requirements should be provided by the bottle vendor to the bottle purchaser with each container lot. Documentation should include a signed and dated cover statement affirming that all QA/QC criteria were met. Copies of raw data from applicable analyses of the QC containers, laboratory standards, check samples, and blanks should be available and provided upon request. Original documentation should be retained for at least 10 years. Minimum documentation that should be available, if applicable, for each lot of containers includes:

- A statement that "Sample container lot _____ meets or exceeds all QA/QC criteria established in 'Specifications and Guidance for Contaminant-Free Sample Containers;'"
- Reconstructed Ion Chromatographs (RICs) from volatile and semivolatile organics determinations, including calibration verification standards, check samples, and blanks;
- GC chromatographs from pesticides determinations, including calibration verification standards, check samples, and blanks;
- ICP, hydride-ICP, or ICP-MS instrument readouts from metals determinations, including calibration verification standards, check samples, and blanks;
- AA raw data sheets and instrument readouts from metals determinations, including calibration verification standards, check samples, and blanks; and
- Cyanide, fluoride, and nitrate/nitrite raw data sheets and instrument readouts from these determinations, including calibration verification standards, check samples, and blanks.

Prior to the first shipment of containers, and at least annually thereafter, the bottle vendor should demonstrate its ability to meet the CRDLs and CRQLs, and establish the reproducibility of the cleaning techniques for each bottle type. The ability to meet the CRDLs and CRQLs is accomplished through the determination of instrument detection limits (IDLs). The bottle vendor should use the procedures in the current CLP Low Concentration Inorganics and Organics SOWs to determine IDLs. IDLs should be below the CRDLs or CRQLs. To establish the reproducibility for each bottle type, the bottle vendor should randomly pick seven containers from a cleaned lot and analyze as described in the Quality Control Analysis part of this Section. Parameter concentrations should be at or below the CRDL or CRQL for each bottle type. Documentation from these analyses should be available and provided upon request.

1. Incoming Materials Inspection:

A representative item from each case of containers should be checked for conformance with specifications provided in Section II. Any deviation should be considered unacceptable. A log of incoming shipments should be maintained to identify material type, purchase order number, and delivery date. The date of incoming inspection and acceptance or rejection of the material should also be recorded on this log.

2. Quality Control Inspection of Cleaned Lots of Containers:

Following container cleaning and labeling, containers should be randomly selected from each container lot to be used for QC purposes. The two categories of QC containers should be as follows:

a. Analysis QC Containers:

One percent of the total number of containers in each lot should be designated as the analysis QC container(s). For lots of less than 100 containers, one container should be designated as the analysis QC container. The sample container preparer should analyze the analysis QC container(s) to check for contamination prior to releasing the associated container lot for shipment. The QC analyses procedures specified in the Quality Control Analysis part of this Section for determining the presence of semivolatile and volatile organics, pesticides, metals, cyanide, fluoride, and nitrate/nitrite should be utilized.

For each analysis QC container(s), an appropriate QC number should be assigned that cross-references the QC container to the related lot of containers. For example, the QC number could be a seven-digit number sequentially assigned to each lot that has undergone QC analysis. Under this numbering scheme, the first alphabetical character would be the container type letter from Figure 1, the next four digits would be assigned sequentially in numerical order starting with "0001" for the first lot to undergo QC analyses, the sixth character would indicate the number of QC container for the lot, (e.g., "1' for the first QC container in the lot, "2" for the second, etc.) and the last character would be either a "C" to indicate clearance or an "R" to indicate rejection.

If the representative analysis QC container(s) passes QC inspection, the related lot of containers should be released, and the appropriate QC number should be entered in the preparation/QC log to indicate clearance of the lot for shipment.

If the analysis QC container(s) are found to be contaminated per the specified QC analysis procedures, the appropriate QC rejection number should be assigned and entered in the preparation/QC log. Any container labels should be removed and the entire lot returned for reprocessing under a new lot number. Excessive QC rejection for a particular container type should be noted for future reference.

A laboratory standard, check sample, and a blank should be run with each QC analysis. A calibration verification standard should be analyzed once every 12 hours. All QC analysis results should be kept in chronological order by QC report number in a central QC file. The QC numbers assigned should be documented in the preparation/QC log, indicating acceptance or rejection and date of analysis.

A container lot should not be released for shipment prior to QC analysis and clearance. Once the containers have passed QC inspection, the containers should be stored in a contaminant-free area until packaging and shipment.

b. Storage QC Containers:

One QC container per lot should be designated as the storage QC container. The storage QC container should be separated from the lot after cleaning and labeling and should be stored in a designated contaminant-free area for one year. The date the container is placed in the storage area should be recorded in the storage QC container log.

If contamination of the particular container lot comes into question at any time following shipment, the storage QC container should be removed from the storage area and analyzed using the QC analysis procedures for that container type (see Quality Control Analysis, this Section). Upon removal, containers should be logged out of the storage area.

The designated storage area should be monitored continuously for volatile contaminants in the following manner. A precleaned, 40-mL vial that has passed a QC inspection should be filled with ASTM Type I organic-free water and be placed in the storage area. This vial should be changed at one-week intervals. The removed vial should be subjected to analysis for volatile organics as described in the Quality Control Analysis part of this Section. Any peaks indicate contamination. Identify contaminants, if present, and include the results in a report to all clients who purchased bottles from the affected lot(s).

B. Quality Control Analysis

The types of QC analyses correlate with the types of containers being analyzed and their future use in sample collection. The QC analyses are intended for the determination of:

- Semivolatile organics and pesticides;
- Volatile organics;
- Metals;
- Cyanide;
- Fluoride; and
- Nitrate/Nitrite.

QC analyses should be performed according to the container type and related sample type and utilize the specific method(s) described below.

1. Determination of Semivolatile Organics and Pesticides:

Container Types: A, E, F, G, H, J, and K

a. Sample Preparation:

- Add 60 mL of pesticide-grade methylene chloride to the container and shake for two minutes.

- Transfer the solvent to a Kuderna-Danish (KD) apparatus equipped with a three-ball Snyder column. Concentrate to less than 10 mL on a steam bath. Split the solvent into two 5 mL fractions for semivolatile and pesticide determinations.

- Add 50 mL of pesticide-grade hexane (for pesticide determinations only) to the KD apparatus by slowly pouring down through the Snyder column. Concentrate to less than 10 mL to effect solvent replacement of hexane for methylene chloride.

- Concentrate the solvent to 1 mL using a micro-Snyder column.

- Prepare a solvent blank by adding 60 mL of the rinse solvent used in step "g" of the cleaning procedure for container types A, E, F, G, H, J, and K (Section III page 14) directly to a KD apparatus, and proceed as above.

b. Semivolatile Organics Sample Analysis:

- Instrument calibration should be performed as described in the most recent CLP Low Concentration Organics SOW with the following exceptions:

  (1) If problems are encountered meeting the %RSD criteria on the initial calibration for semivolatiles, the high concentration point should be deleted and a four-point calibration used.

  (2) The low concentration standard should be used for the continuing calibration standard for semivolatile analyses.

  (3) The percent difference window should be widened to ± 30 percent for all compounds.

- Inject 1 μL of solvent into a gas chromatograph/mass spectrometer (GC/MS).

- Calibration verification standards should be analyzed as described in the most recent CLP Low Concentration Organics SOW.

- Blanks should be run as described in the most recent CLP Low Concentration Organics SOW.

- If compounds other than those listed in Table 2 are found in the container blank that are not in the solvent blank at a peak height or peak area greater than 20 percent of the nearest internal standard, the containers should be rejected (See Section II, Table 2 for compound specifications).

- Identify and quantitate any contaminant(s) that cause rejection of a container lot.

- A standard mixture of the nine semivolatile organic compounds listed in Table 3 (page 26) with concentrations in the 5-20 ppb range should be analyzed to ensure that sensitivities are achieved that will meet contract required quantitation limits. This standard should be prepared from a different source from the calibration standards.

c. Pesticides Sample Analysis:

- Instrument calibration should be performed as described in the most recent CLP Low Concentration Organics SOW.

- Inject 1 μL of solvent into a gas chromatograph (GC) equipped with an electron capture detector (ECD).

- Calibration verification standards should be analyzed as described in the most recent CLP Low Concentration Organics SOW.

- Blanks should be run as described in the most recent CLP Low Concentration Organics SOW.

- If compound peaks other than those listed in Table 2 are at a peak height or peak area greater than 5 percent of the peak height or peak area of tetra chloro-m-xylene, the containers should be rejected (See Section II, Table 2).

- Identify and quantitate any contaminant(s) that cause rejection of a container lot.

- A standard mixture of the seven pesticide compounds listed in Table 3 (page 26) with concentrations in the 0.01 to 1 ppb range should be analyzed to ensure that sensitivities are achieved that will meet contract required quantitation limits. This standard should be prepared from a different source from the calibration standards.

2. Determination of Volatile Organics:

Container Types: B and D

a. Sample Preparation:

- Fill the container with ASTM Type I organic-free water.

- Cap the container and let stand for 48 hours.

b. Sample Analysis:

- Instrument calibration should be performed as described in the most recent CLP Low Concentration Organics SOW with the following exceptions:

(1) If problems are encountered meeting the %RSD criteria on the initial calibration for volatiles, the high concentration point should be deleted and a four-point calibration used.

(2) The low concentration standard should be used for the continuing calibration standard for volatile analyses.

(3) The percent difference window should be widened to $\pm$ 30 percent.

- Calibration verification standards should be analyzed as described in the most recent CLP Low Concentration Organics SOW.

- Blanks should be run as described in the most recent CLP Low Concentration Organics SOW. The blank should consist of an aliquot of the ASTM Type I water used in the sample preparation.

- If compounds other than those listed in Table 2 are found in the container blank that are not in the solvent blank at a peak height or peak area greater than 20 percent of the nearest internal standard, the containers should be rejected (See Section II, Table 2 for compound specifications).

- Identify and quantitate any contaminant(s) that cause rejection of a container lot.

- A standard mixture of the five volatile organic compounds listed in Table 3 (page 26) with concentrations in the 1-5 ppb range should be analyzed to ensure that sensitivities are achieved that will meet contract required quantitation limits. This standard should be prepared from a different source from the calibration standards.

3. Determination of Metals:

Container Types: A, C, E, F, G, H, J, K and L

a. Sample Preparation:

- Add 100 mL of ASTM Type I deionized water to the container, and acidify with 1.0 mL of reagent-grade HNO_3. Cap and shake for three to five minutes.

- Cap the container and let stand for 48 hours.

- Treat the sample as a dissolved metals sample. Analyze the undigested water using the most recent CLP Low Concentration Inorganics SOW.

b. Sample Analysis:

- Instruments used for the analysis of the samples should meet the contract required detection limits in Table 1.

- The ASTM Type I deionized water should be analyzed before use on the bottles that are designated for analysis to ensure that contaminated water is not used for rinsing the bottles.

- Calibration verification standards should be analyzed as described in the most recent CLP Low Concentration Inorganics SOW.

- Blanks should be analyzed as described in the most recent CLP Low Concentration Inorganics SOW. A calibration blank is a solution made up exactly like the sample preparation solution. The calibration blank should be less than the values contained in Table 1.

- A set of standards in the expected working range should be analyzed with each analytical run. The acid matrix of the standards, blank, and quality control samples should match that of the samples.

- Concentrations at or above the detection limit for each parameter (listed in Table 1) should be cause for rejection of the lot of containers. **NOTE:** The sodium detection limit for container types A, E, F, G, H, J, and K is 5000 μg/L unless the containers will be used for low concentration analyses, then the detection limit is 500 μg/L.

4. Determination of Cyanide:

Container Types: A, C, E, F, G, H, J, K and L

a. Sample Preparation:

- Place 250 mL of ASTM Type I deionized water in the container. Add 1.25 mL of 6*N* NaOH (for container types F and G use 100 mL of ASTM Type I deionized water and 0.5 mL of 6*N* NaOH). Cap the container and shake vigorously for two minutes.

b. Sample Analysis:

- Analyze an aliquot as described in the most recent CLP Low Concentration Inorganics SOW.

- The detection limit should be 10 μg/L or lower.

- Calibration verification standards should be analyzed as described in the most recent CLP Low Concentration Inorganics SOW.

- Blanks should be run as described in the most recent CLP Low Concentration Inorganics SOW. The calibration blank should consist of an aliquot of the ASTM Type I water used above.

- A set of standards in the expected working range, a check sample, and blank should be prepared exactly as the sample was prepared.

- The detection of 10 μg/L cyanide (or greater) should be cause for rejection of the lot of containers. **NOTE:** Contamination could be due to the container, the cap, or the NaOH.

5. Determination of Fluoride:

Container Types: A, C, E, F, G, H, J, K and L

a. Sample Preparation:

- Place 250 mL of ASTM Type I deionized water in the container (for container types F and G use 100 mL of ASTM Type I deionized water). Cap the container and shake vigorously for two minutes.

b. Sample Analysis:

- Analyze an aliquot as described in the most recent CLP Low Concentration Inorganics SOW.

- The detection limit should be 200 μg/L or lower.

- Calibration verification standards should be analyzed as described in the most recent CLP Low Concentration Inorganics SOW.

- Blanks should be run as described in the most recent CLP Low Concentration Inorganics SOW. The calibration blank should consist of an aliquot of the ASTM Type I water used above.

- A set of standards in the expected working range, a check sample, and blank should be prepared exactly as the sample was prepared.

- The detection of 200 µg/L (or greater) of fluoride should be cause for rejection of the lot of containers. **NOTE:** Contamination could be due to the container or the cap.

6. Determination of Nitrate/Nitrite:

Container Types: A, C, E, F, G, H, J, K and L

a. Sample Preparation:

- Place 250 mL of ASTM Type I deionized water in the container (for container types F and G use 100 mL of ASTM Type I deionized water). Cap the container and shake vigorously for two minutes.

b. Sample Analysis:

- Analyze an aliquot as described in the most recent CLP Low Concentration Inorganics SOW.

- The detection limit should be 100 µg/L or lower.

- Calibration verification standards should be analyzed as described in the most recent CLP Low Concentration Inorganics SOW.

- Blanks should be run as described in the most recent CLP Low Concentration Inorganics SOW. The calibration blank should consist of an aliquot of the ASTM Type I water used above.

- A set of standards in the expected working range, a quality control sample, and blank should be prepared exactly as the sample was prepared.

- The detection of 100 µg/L (or greater) of nitrate/nitrite should be cause for rejection of the lot of containers. **NOTE:** Contamination could be due to the container or the cap.

C. Preparation and Labeling

Sampling for environmental specimens requires that sample containers be transported to field sites prior to sample collection. As a result, considerable time may elapse between the receipt of sample containers and collection of the samples. Because of the large number of samples taken at any one site, accounting for all sample containers can become extremely difficult. The following guidance on the identification and tracking of sample containers is based on procedures that have been used successfully in the CLP bottle program.

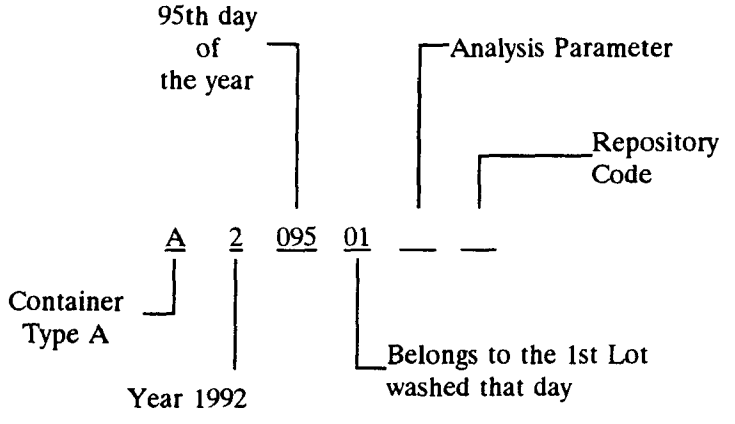
1. Each shipment should be inspected to verify that the requested number of cleaned and prepared sample containers have been supplied and meet the requirements specified in Section II (Tables 1 and 2). If any shipment fails to meet the required specifications, it should be discarded and replaced with a supply of sample containers that meet the required criteria.

2. The sample containers should be removed and prepared in accordance with the methods designated below.

3. A permanent nine-digit lot number should be assigned to each lot of sample containers for identification and tracking purposes throughout the life of the containers. Figure 2 provides an example of a lot number sequence.

FIGURE 2

LOT NUMBER SEQUENCE



a. The first digit represents the container type in Section II (Figure 1).

b. The second digit represents the last digit of the calendar year.

c. The next three digits represents the day of the year on which the sample containers were washed.

d. The sixth and seventh digits represent the daily lot number.

e. The eighth digit represents the analysis parameter where:

A = Semivolatile organics, pesticides, metals, cyanide, and fluoride;
B = Metals, cyanide, and fluoride;
V = Volatile organics;
S = Semivolatile organics and/or pesticides;
M = Metals;
C = Cyanide;
F = Fluoride; and
N = Nitrate/nitrite.

f. The final digit represents the identification of the person who prepared the lot.

4. The lot number for each container should be entered, along with the date of washing, type of container, and number of containers per lot, into the preparation/QC log book.

5. Lot numbers printed with solvent resistant ink on a nonremovable label should remain with the corresponding containers throughout the cleaning procedure.

6. After sample container cleaning and drying, the label should be affixed to the containers in a permanent manner.

7. At least one face should be clearly marked, excluding the top and bottom faces, of each case of sample containers with the assigned lot numbers.

TABLE 3

STANDARD MIXTURES OF ORGANIC COMPOUNDS TO VERIFY SENSITIVITY

Volatiles	Semivolatiles	Pesticides
Methylene Chloride	Nitrobenzene	Gamma-BHC
Acetone	4-Chloroaniline	Heptachlor
2-Butanone	2,6-Dinitrotoluene	Aldrin
Trichloroethene	Diethylphthalate	Dieldrin
Toluene	4-Bromophenyl-phenylether	Endrin
	Hexachlorobenzene	4,4'-DDT
	Pentachlorophenol	Aroclor 1260
	Di-n-butylphthalate	
	bis(2-Ethylhexyl)phthalate	

*U.S. G.P.O.:1993-341-835:81056