

**CL-055R**

**SAMPLING  
LIQUID WASTES**

**Revision: 5**

**Date Effective: September 2017**

**Dugway Proving Ground EPA ID Number: UT3750211259**

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## 1.0 Scope and Application

This method provides procedures to collect liquid waste samples regulated by the regulatory compliance program at Dugway Proving Ground (DPG). This method also applies to liquid samples that have been combined with solids (such as vials, gloves, towels, etc.) for decontamination purposes.

General quality control (QC) guidelines for sampling, sampling equipment, and chain-of-custody (COC) are found in the DPG Waste Permit, Attachment 1-10, *Central Hazardous Waste Storage Facility (CHWSF) Quality Assurance Program Plan (QAPP)*. A method schematic is provided in Figure 1.

## 2.0 Scientific Basis

Because liquid waste samples may be heterogeneous, it is important to collect representative samples. In addition, sampling should minimize sample loss and degradation and provide sufficient sample volume for laboratory analysis. The Composite Liquid Waste Sampler (COLIWASA) is used to collect free-flowing liquids and slurries from drums, shallow open tanks, pits, etc.

## 3.0 Terminology

This section lists in alphabetical order all terms, abbreviations, and acronyms unique to understanding this method.

- CAS<sup>®</sup> – Chemical Abstracts Service<sup>®</sup>
- Chemical Agent – Any of several highly toxic chemical compounds (including GA, GB, GD, GF, HD, HN1, HN3, Lewisite, HT, T, and VX) that are intended for use in military operations
- COC – Chain-of-Custody
- COLIWASA – Composite Liquid Waste Sampler
- Decontamination – The process of decreasing the amount of chemical agent on any person, object, or area by absorbing, neutralizing, destroying, ventilating, or removing chemical agents
- CTD – Chemical Test Division
- DPG – US Army Dugway Proving Ground
- Field Duplicate – Duplicate samples collected in the field to establish the overall precision of the sampling and analytical process. Duplicates are required when new or unknown waste sources are collected and are handled like routine samples in the laboratory.
- GA – tabun, ethyl N,N-dimethylphosphoroamidocyanidate (CAS<sup>®</sup> No. 77-81-6)
- GB – sarin, isopropyl methylphosphonofluoridate (CAS<sup>®</sup> No. 107-44-8)
- GD – soman, pinacolyl methylphosphonofluoridate (CAS<sup>®</sup> No. 96-64-0)
- GF – cyclohexyl methylphosphonofluoridate (CAS<sup>®</sup> No. 329-99-7)
- HD – distilled mustard, bis-2-chloroethyl sulfide (CAS<sup>®</sup> No. 505-60-2), a blister agent.
- HN1 – bis (2-chloroethyl) ethylamine (CAS<sup>®</sup> No. 538-07-8), a blister agent.
- HN3 – tris-2-chloroethylamine (CAS<sup>®</sup> No. 555-77-1), a blister agent.
- Lewisite – dichloro-(2-chlorovinyl)arsine (CAS<sup>®</sup> No. 541-25-3), a blister agent.

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- LIMS – Laboratory Information Management System
- mL – milliliters(s)
- PPE – personal protective equipment
- PVC – polyvinyl chloride
- QAPP – Quality Assurance Program Plan
- QC – quality control
- Rinse blank – A sample collected in the field to demonstrate that no cross-contamination has occurred during sampling. One rinse blank per field sample lot is needed when nondisposable sampling equipment is used. Rinse blanks are not required when disposable sampling equipment is used.
- Sample collection lot – Twenty or fewer samples collected from the same waste description at one time (shift) by a single team of sampling personnel. Each field sample lot for liquid is accompanied by field QC samples including a field duplicate and an equipment rinse blank when using nondisposable sampling equipment.
- T – bis[2-(2-chloroethylthio)ethyl]ether (CAS<sup>®</sup> No. 6391-89-8)
- VX – o-ethyl s-(2-diisopropylaminoethyl) methylphosphonothioate (CAS<sup>®</sup> No. 50782-69-9) a blister agent.

#### 4.0 Safety

Generally, regulatory compliance samples have been exposed to chemical agent and subsequently decontaminated or contain other hazardous substances. Handle all samples with caution. For all operations involving chemical agents, comply with all US Army safety rules and regulations. Be familiar with and follow safety guidelines contained in Material Safety Data Sheets for the chemicals being used or sampled.

Sample-collection personnel performing this procedure will be trained in the use of personal protective equipment (PPE).

Before beginning sampling, sample-collection personnel will fully understand the waste to be sampled and take appropriate safety precautions. Exercise caution when opening drums or other sealed containers. Wear the following minimum PPE: gloves, a smock or coveralls, and an appropriate respirator.

Obtain appropriate clearances before entering restricted areas. Transport samples using only government- or contractor-owned vehicles. Do not transport samples in private vehicles.

#### 5.0 Apparatus and Reagents

To collect liquid waste samples, the following items may be required:

- Ice chest with ice or blue-ice packs
- Sampling logbook
- COC/Analysis Request form
- Clean, disposable, glass COLIWASA
- Clear glass sample containers with Teflon<sup>®</sup>-lined lids
- Deionized water

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- Labels for sample container
- PPE

When possible, use disposable COLIWASAs. Reusable COLIWASAs can be used if they are thoroughly cleaned before use.

## 6.0 Standards and QC

Field QC samples are intended to measure the cleanliness and representativeness of the sampling activities. Sample-collection personnel are responsible for correctly collecting field QC samples. Field duplicates are required when new or unknown waste sources are collected. Sample-collection personnel will collect field duplicates in the same manner as the other samples in the sample collection lot. Collect one rinse blank per sample collection lot when nondisposable sample equipment is used. Rinse blanks are not required when disposable sampling equipment is used.

## 7.0 Procedure

To document sample collection, sample collection personnel perform the procedures in Paragraph 7.1. To sample liquid wastes from drums and tanks, perform the procedures in Paragraphs 7.2 and 7.3, respectively.

### 7.1 Documenting Sample Collection

To document sample collection, sample-collection personnel perform the following tasks:

- Record the following information related to sample collection as it occurs using a field logbook or worksheet:
  - Sample collection personnel
  - Sample collection date
  - Collection time for each sample
  - Location of sampled material
  - Sample identification (i.e., drum number, barcode number, etc.)
  - Description of the material sampled, if applicable (i.e., background or historical information, description of phases, etc.)
  - Identifying marks or numbers on the sample container, if any
  - Sample collection method and description
  - PPE worn
  - Unusual or hazardous conditions
  - Other observations
- Complete the COC/Analysis Request form (see the QAPP for the Analysis of Chemical Agent-Related Waste) before submitting samples to the laboratory. The information on the COC/Analysis Request form should be consistent with the information recorded in the field records. Mark the COC/Analysis Request form to indicate which analytes are to be determined and note unusual or potentially hazardous conditions.

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## 7.2 Sampling Drums

To collect liquid or combined liquid/solid waste samples in drums or other similar containers, sample-collection personnel perform the following tasks:

1. Before beginning the sampling operation, ensure that all sample-collection personnel and observers are wearing appropriate PPE.
2. Visually inspect container for signs of deterioration, pressure build-up, or other conditions detrimental to sampling. Consult with management before attempting to open damaged containers. If the material to be sampled has been disturbed before sampling, allow time for the contents to separate into their representative phases.
3. If the COLIWASA sampler has been previously used, collect an equipment rinse blank by filling the COLIWASA from the top with deionized water and discharging 100 milliliters (mL) into a sample bottle.
4. Label the sample bottle with the following information:
  - Sample field identification number
  - Name of collector
  - Date of collection
  - Time of collection
  - Place of collection
  - Analyses requested
  - Comments including any unusual or hazardous conditions
5. To obtain a sample, open the waste container slowly, allowing the contents to vent if necessary.
6. Slowly immerse the COLIWASA into the waste. Ensure that the level of the liquid in the tube remains even or nearly even with the surface of the liquid outside the tube. For liquid samples that have been combined with solids for decontamination purposes, only the liquid is sampled.

**NOTE:** A polyvinyl chloride (PVC) sampling screen may be used to aid in the sampling of liquid/solid combination waste in drums. Before sampling, ensure that the sampling screen is standing straight and touching the bottom of the drum.

7. Close the COLIWASA and slowly remove it from the waste.
8. If the sample appears to be a single phase, drain the liquid into a clean sample bottle. Collect a minimum volume of 100 mL of sample. If necessary, resample until 100 mL has been collected.
9. If a sample appears to have a distinct organic solvent layer, two representative samples need to be collected. The determination of phase height and if the solvent layer is greater than or equal to 10% of the total sample volume is performed at the time of analysis, but

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could also be done at the time of sampling. The determination of the solvent percentage is described in method CL002R. Alternative techniques may also be employed to determine phase height such as using graduated sample jars or other glassware. If the sampling team is unsure of the exact percentage of the solvent layer, they should collect two samples as a precaution and log both samples into the Laboratory Information Management System (LIMS). In the event that only one sample is needed for analysis, the second sample may be cancelled. Slowly immerse the COLIWASA into the waste with the stopper open. Ensure that the level of the liquid in the tube remains even or nearly even with the surface of the liquid outside the tube and close the stopper. Drain the entire contents of the COLIWASA into the sample bottle. No separation of the phases is performed at this time. Ensure that a minimum of 100 mL is collected for each sample.

10. Obtain a duplicate from at least one sample in twenty or fewer in the field sample collection lot if this sample is from a new or unknown waste source.
11. Label the sample bottle with the following information:
  - Sample field identification number
  - Name of collector
  - Date of collection
  - Time of collection
  - Place of collection
  - Analyses requested
  - Comments including any unusual or hazardous conditions
12. Wearing cut/puncture-resistant gloves, carefully break used, disposable COLIWASAs into the original waste container, or properly treat them as chemical agent-related waste.
13. Dispose of any contaminated gloves, paper towels, or other sampling materials in the waste container.
14. Reseal the waste container.
15. Place the samples in an ice chest on ice or blue-ice packs.
16. Immediately transport the samples and COC/Analysis Request form to the laboratory under COC procedures as described in the QAPP

### 7.3 Sampling Tank Contents

Generally, one sample is collected per drum or container of liquid waste. In the case of homogeneous liquid wastes being transferred from a large storage tank (>500 gallons) to multiple 55-gallon drums (in a single batch), two samples (one at the beginning and another at the end of the transfer process) are considered sufficient. If the waste stream has multiple layers or non-homogeneous waste, the number of samples to be collected will be agreed upon with the Division of Solid and Hazardous Waste. A rinse blank is collected if the sample collection equipment has been previously used. The test sample is usually obtained at the time the tank contents are

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transferred to 55-gallon drums. This procedure assumes that the tank liquids are a single phase and have been thoroughly mixed. If the tank contents are not a single phase and/or thoroughly mixed, collect individual samples from the drums after transfer as described in Section 7.2.

To collect liquid waste samples in tanks or other large containers, sample-collection personnel will perform the following tasks:

1. Before beginning the sampling operation, ensure that all sample-collection personnel and observers are wearing appropriate PPE.
2. Visually inspect the tank for signs of deterioration, pressure build up, or other adverse conditions. Consult with management if adverse conditions exist.
3. Begin the liquid transfer process and allow transfer lines to flush thoroughly.
4. Carefully fill a clean sample bottle with a minimum of 100 mL of sample.
5. Label the sample bottle with the following information:
  - Sample field identification number
  - Name of collector
  - Date of collection
  - Time of collection
  - Place of collection
  - Analyses requested
  - Comments including any unusual or hazardous conditions
6. Obtain a field duplicate sample towards the end of the sample transfer process in the same manner as described in this section for field samples.
7. Place the samples in an ice chest on ice or blue-ice packs.
8. Immediately transport the samples and COC/Analysis Request form to the laboratory under COC procedures as described in the QAPP. Avoid excessive exposure to heat and sunlight.

## **8.0 Data Reduction and Assessment**

The relative percent difference between duplicate samples and the equipment rinse blank results may be related to the sample collection. Inform sample-collection personnel of any problems with these quality indicators to facilitate continuous improvement in the sample collection process.

## **9.0 References**

US Army Dugway Proving Ground (DPG), Utah, Waste Permit, Attachment 1-10, *Central Hazardous Waste Storage Facility (CHWSF) Quality Assurance Program Plan*.

**Figure 1**  
**Method Schematic**

