CL-002R

ANALYSIS OF CHEMICAL AGENTS BY GAS CHROMATOGRAPHY WITH FLAME-PHOTOMETRIC AND/OR MASS SELECTIVE DETECTION

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1.0 Scope and Application

This method provides procedures for analyzing chemical agents Tabun (GA), Sarin (GB), Soman (GD), Cyclohexyl Methylphosphonofluoridate (GF), Mustard (HD), Bis (2-chloroethyl) Ethylamine (HN1), Tris-2-Chloroethylamine (HN3), Lewisite, T, and O-ethyl S-(2-diisopropylaminoethyl (VX) in environmental samples using gas chromatography (GC) with detection using a flame--selective detector (MSD). This method is applicable to analyzing liquids, soils, or other solids regulated by the regulatory compliance program at US Army Dugway Proving Ground (DPG).

General quality control (QC) guidelines for sampling, sampling equipment, and chain-of-custody 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

Liquid or soil samples to be analyzed for chemical agents are first micro-extracted with an appropriate solvent. Soil/solid samples to be analyzed for chemical agents other than Lewisite and VX are extracted using isopropyl alcohol (IPA) and chloroform. Soil/solid samples to be analyzed for VX are extracted with IPA and a solvent mixture of chloroform and 2-(diisopropylamino) ethanol. Soil/solid samples to be analyzed for Lewisite are extracted with IPA and a solvent mixture of chloroform and 1,2-ethanedithiol. The solvent mixtures and co-solvents aid in the extraction of chemical agents from soils/solids, particularly moist soils. In the case of Lewisite, the 1,2-ethanedithiol derivatizes the Lewisite, converting it into a chromatographable compound. Liquids are extracted with the following solvents: Agents other than Lewisite and VX are extracted with chloroform: Lewisite is extracted with a solvent mixture of chloroform and 1,2-ethanedithiol, and VX is extracted with a solvent mixture of chloroform and 2-(diisopropylamino) ethanol.

After extraction, components of the extract are separated by traditional GC techniques. Detection is achieved using an FPD equipped with the appropriate optical bandpass filters or an MSD in selected-ion mode (SIM) or full-scan mode. HN1, HN3, and Lewisite are analyzed using only the MSD. Identification by FPD analysis is predicated upon three, independent criteria: solvent extractability, GC retention time, and sulfur or phosphorus content. For MSD analysis, identification is predicated upon solvent extractability, retention time, and ion abundance (spectral matching).

Potential positive interferences are possible because of other sulfur or phosphorus-containing compounds, such as pesticides, or other organic compounds. Negative interferences are possible when analyzing samples with high levels of hydrocarbons, such as gasoline or oil. These interferences are not expected when analyzing routine liquids or soils.

3.0 Terminology

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

- %R – percent recovery

- Calibration check (CC) standard – A mid-range analytical standard run in a specified sequence or time interval to verify that the calibration of the analytical system remains in control. The ICV standard solution or one of the standards used for the initial calibration could be used. See CCV below.
- CAS® – chemical abstracts service®
- Continuing-calibration verification (CCV) – A sample of known concentration analyzed every 10 samples or at the end of the sequence of analysis to verify that the calibration curve is still viable. The CCV can be one of the calibration standards or the ICV.
- Chemical agent – Any of several highly toxic chemical compounds (including GA, GB, GD, GF, HD, HN1, HN3, Lewisite, T, and VX) intended for use in military operations.
- CWA – chemical warfare agent
- Decontamination (decon) – The process of decreasing the amount of chemical agent on any person, object, or area by absorbing, neutralizing, destroying, ventilating, or removing chemical agents.
- DPG – US Army Dugway Proving Ground
- EDT – 1,2-Ethanedithiol
- FPD – flame photometric detector
- GA – tabun, ethyl N,N-dimethylphosphoroamidocyanidate (CAS® No. 77-81-6)
- GB – sarin, isopropyl methylphosphonofluoridate (CAS® No. 107-44-8)
- GC – gas chromatography/gas chromatograph
- GD – soman, pinacolyl methylphosphonofluoridate (CAS® No. 96-64-0)
- GF – cyclohexyl methylphosphonofluoridate (CAS® No. 329-99-7)
- HD – mustard, distilled, bis-2-chloroethyl sulfide (CAS® No. 505-60-2), a blister agent.
- HN1 – bis (2-chloroethyl) ethylamine (CAS® No. 538-07-8), a nitrogen mustard
- HN3 – tris-2-chloroethylamine (CAS® No. 555-77-1), a nitrogen mustard
- HPLC – high-performance liquid chromatography
- IAW – in accordance with
- ID – identification
- Initial calibration – A mathematical model of the response of the detector to varying concentrations of analyte. The initial calibration is determined by plotting the intensity of detector response versus the known concentration of multiple standards. The calibration curve is used to quantitate the unknown concentrations of analyte in field and QC samples.
- Initial-calibration verification (ICV) standard – A standard material, prepared independently from calibration standards, that is used to verify the accuracy of initial calibration standards.
- IPA – isopropyl alcohol
- Lewisite – 2-chlorovinyldichloroarsine (CAS® No. 541-25-3)
- LIMS – Laboratory Information Management System
- Matrix spike/matrix spike duplicate – A positive control prepared in duplicate from a field sample to establish the effect of the matrix on precision and accuracy.
- Method blank (MB) – A negative control prepared in the laboratory to establish that the overall analytical system is not causing significant interference with target analyte detection and quantitation.
- Method blank spike (MBS) – A positive control prepared in the laboratory to establish that the overall analytical system is performing within expected tolerances with respect to the analytical system's ability to accurately measure target concentrations in the absence

of undue matrix effects.

- Method blank spike duplicate (MBSD) – A positive control prepared in the laboratory to establish that the overall analytical system is performing within expected tolerances with respect to the analytical system's ability to precisely measure target concentrations in the absence of undue matrix effects.
- Method detection limit (MDL) – An estimate of the lowest level of an analyte that a method can distinguish from background noise.
- MS – mass spectrometer
- MSD – mass selective detector
- NA – not applicable
- NIST – National Institute of Standards and Technology
- QA – quality assurance
- QAPP – Quality Assurance Program Plan
- QC – quality control
- r^2 – correlation coefficient squared
- Reporting limit (RL) – The limit at which a number can be reasonably considered to be quantitatively accurate. This number is derived by measuring the method detection limit, multiplying by four, and rounding to a convenient number.
- RPM – revolutions per minute
- RSD – relative standard deviation
- SARM – Standard Analytical Reference Material
- SDS – safety data sheet
- SIM – selected-ion mode
- SOP – standing operating procedure
- SR – sample result (unspiked)
- SSR – spiked sample result
- STD – standard
- T – bis (2-chloroethylthioethyl) ether (CAS® No. 63918-89-8) a chemical agent similar to HD.
- VX – o-ethyl S-(2-diisopropylaminoethyl) methylphosphonothioate (CAS® No. 50782-69-9) a persistent-nerve agent.

4.0 Safety

Generally, regulatory compliance samples received by the laboratory have been or are suspected of being exposed to chemical warfare agent (CWA) and subsequently decontaminated. Handle all samples with caution. For all operations involving chemical agents, comply with all laboratory safety rules and regulations. Be familiar with and follow safety guidelines contained in safety data sheets for the chemicals being used or analyzed.

5.0 Apparatus and Reagents

To perform the procedures in this method, the following apparatus and reagents may be required:

5.1 Apparatus

Ensure that the following apparatus are available to perform this method:

- Temperature-programmable GC instrument equipped with an FPD (with appropriate optical filters) or MSD, depending on which analytes are to be determined and/or the availability of instrumentation.
- DB-5 Column, 30 m x 0.32 mm or 30 m x 0.25 mm (or equivalent) with a film thickness of 0.25 to 0.5 µm. Other columns may be used if it is demonstrated that suitable results can be obtained while using them. For example, if analyte confirmation is to be performed on a GC/FPD, another column must be used which preferably elutes the analytes in a different order than the DB-5 column.
- Computer equipped with appropriate software for analyzing chromatographic data or another, appropriate, data collection device.
- Auto-sampler
- Analytical balance
- Graduated pipettes or automated pipettor
- 40- to 50- mL Centrifuge tubes with caps
- Sampler vials with Teflon® lined caps, 2 mL
- Vial rack
- Vortex mixer (optional)
- Broad-range pH-indicating paper
- Glass barrel micro-syringes
- Disposable pipettes
- Separatory funnels
- Automatic diluter
- Centrifuge

5.2 Reagents

Obtain the following reagents to perform this method:

- Deionized water
- Chloroform, pesticide-grade, high-performance liquid chromatography (HPLC)-grade, or equivalent
- Ultra-pure or equivalent (hydrocarbon free) gases (helium, argon, or nitrogen)
- Sodium chloride, reagent-grade or better
- Anhydrous, sodium sulfate, reagent-grade or better
- IPA, pesticide-grade, HPLC-grade, or equivalent
- 2-(Diisopropylamino)ethanol, reagent-grade, if possible, otherwise highest purity available
- 1,2-Ethanedithiol (EDT), reagent-grade, if possible, otherwise highest purity available

Using the apparatus and reagents listed in Paragraphs 5.1 and 5.2 technical personnel prepare the following solutions:

- Brine reagent solution, 5% (weight/volume) – Dissolve 50 g of sodium chloride in 1.0 L of deionized water.

- 2-(Diisopropylamino)ethanol/chloroform mixture – Mix 40 g (approximately 48 mL) of 2-(diisopropylamino) ethanol with 10 g of reagent-grade water, and 2.0 L of chloroform. Shake the mixture until all components are mixed. Other quantities may be prepared to meet sample demands. Keep proportions constant.
- 1,2-Ethanedithiol/Chloroform mixture – To prepare approximately 2 L, mix 20 mL of EDT Chloroform to make 2 liters. Other quantities may be prepared to meet demand. Keep the proportions constant.
- Unless otherwise stated, reagent solutions may be used for up to 6 months or until signs of degradation are noted (such as cloudiness or a change in pH). Record the following information about the preparation of each solution in the analyst's notebook or in the laboratory Information management system (LIMS):

    - Analyst's initials
    - Date of preparation
    - Source reagent's name, manufacturer, and lot number
    - Source reagent's concentration (if applicable)
    - Source reagent's mass or volume
    - Solvent's name, grade, manufacturer, and lot or bar code number
    - Amount of solvent used or final volume achieved
    - Final concentration
    - Expiration date

6.0 Standards and Quality Control

This section presents procedures for technical personnel to prepare standards and laboratory QC samples for chemical agents in liquids, soils, or other solids analyzed by GC.

6.1 Preparing Standards

Technical personnel will prepare all stock, initial-calibration, and verification standards, as well as spiking solutions as required by the procedures in this method. Technical personnel will document the preparation of all standards in the logbook or the LIMS.

6.1.1 Stock Standards

Stock standards are prepared in accordance with (IAW) approved procedures.

6.1.2 Initial Calibration Standards

To prepare initial-calibration standards, perform the following steps:

1. Prepare initial-calibration standards of at least five concentrations in the approximate range shown in Table 1. The concentration of one of the standards should be at or below the action limit.

Table 1: Initial Calibration Standard Concentration Ranges

Chemical Agent	Concentration Range (mg/L)
GA, GB, GD, GF, VX, HD, HN1, HN3, Lewisite, T	One of the standards at or below the action limit with the other standards spaced across the desired working range, with a minimum of 5 standards.

2. Prepare the standards using the same solvent that will be used to extract samples.
3. Combine the standards into different mixes so that VX and HD are in different solutions. Lewisite should also be in a separate solution.
4. Prepare initial calibration standards using volumes that are easily measured. Calculate the resulting concentration to at least two significant figures. An example of initial-calibration standard preparation is shown in Table 2.
5. As shown in the table, calculate the volume of concentrated stock solution to make 50.00 mL of a 6 μg/mL standard. For example, 0.30 mL of a stock solution at 1,000 μg/mL diluted to 50.00 mL yields a working solution with a resulting concentration of 6 μg/mL (Stock #1).

Table 2: Example of Preparing Initial Calibration Standard Preparation

Source		Dilution Volume (mL)	Resulting Solution	Resulting Concentration (μg/mL)
Diluted Solution	Volume (mL)			
Stock	0.30	50.00	STD[a] 6	6.00
STD 6	5.0	10.00	STD 5	3.00
STD 5	5.00	10.00	STD 4	1.5
STD 4	5.00	10.00	STD 3	0.75
STD 3	5.00	10.00	STD 2	0.375
STD 2	5.00	10.00	STD 1	0.1875

[a]Standard

6. Place each initial calibration standard solution in an appropriate container.
7. Store standard solutions at a temperature of ≤10°C but above freezing.
8. Allow solutions to equilibrate to ambient room temperature for at least 30 minutes before use.
9. Use single-component and multi-component initial-calibration standards for a period not exceeding 30 days.

6.1.3 Verification Standards

To prepare verification standards, perform the following steps:

1. Prepare calibration verification standards at a convenient concentration in the mid-range of the calibration curve. For example, adding 0.300 mls of a stock solution at 1,000 μg/mL to a 50.00-mL volumetric flask, and diluting the mixture to volume will yield a 6 ug/mL solution.
2. Prepare different solutions to keep HD, VX, and Lewisite separate.
3. Use a different concentrated-stock standard than that used to prepare initial-calibration standards. However, if sources of neat agent stocks are limited, a different analyst may prepare the verification standards using the same neat stock solution used to prepare the initial calibration standard. Ensure that the instrument response from the initial calibration standards is within the expected range. Large differences in responses from historical data of standards and other stock solutions may indicate that the standard was prepared incorrectly. If this is the case, solutions will be re-prepared and reanalyzed.
4. Calculate the exact concentration for each analyte.
5. Place each verification standard solution in an appropriate container.
6. Store verification standards at a temperature of ≤10°C but above freezing.
7. Allow solutions to equilibrate to ambient room temperature for at least 30 minutes before use.
8. Use single-component and multi-component verification standards for a period not exceeding 30 days.

6.1.4 Spiking Solutions

To prepare spiking solutions, perform the following steps:

1. Prepare spiking solutions in the same manner as initial-calibration standards with the exception that the solvent will be IPA. Prepare them using the concentration listed in Table 3. For example, prepare spiking solutions by adding 0.300 mls of a 1,000 μg/mL concentrated stock of each chemical agent to a 50.00 mls volumetric flask and filling to volume. The resulting concentration will be approximately 6 μg/mL.

Table 3: Spiking Solution Concentration

Chemical Agent	Concentration (mg/L)
All agents	Approximately 6 (character code 2248)

2. Keep VX, Lewisite and HD in separate solutions.
3. If neat agent stock solutions are not available, make HN1 and HN3 spiking solutions using hydrochloride salts.
4. Calculate the exact concentration of each chemical agent.
5. Use spiking solutions for a period not exceeding 30 days.
6. Store spiking solutions at a temperature of ≤10°C but above freezing.
7. Use spiking solutions at room temperature by allowing them to sit at room temperature for at least 30 minutes before use.

6.2 Preparing Laboratory QC Samples

Technical personnel will prepare method blank (MB), method blank spike (MBS), and method blank spike duplicate (MBSD) samples IAW Table 4. Matrix spike/matrix spike duplicate samples are to be prepared and analyzed for non-active matrix samples (i.e., samples that do not contain a decontaminating agent) at the rate of one in twenty or one per batch if the batch size is less than twenty. Matrix spikes are prepared in the same manner as the MBS/MBSD but use field samples instead of analyte-free material. For each QC sample prepared, technical personnel record the following information in the logbook or Laboratory Information Management System (LIMS):

- Spiking solution identification (ID) number
- Volume of spiking solution used
- Concentration of spike solution used
- Analyst's initials
- Date prepared

Table 4: Quality Control (QC) Sample Preparation

	Laboratory QC Sample		
Matrix	**Method Blank (MB)**	**Matrix Spike (MS)/ Matrix Spike Duplicate (MSD)**	**Method Blank Spike (MBS)/Method Blank Spike Duplicate (MBSD)**
Liquid	Use 30 mL of brine solution as the sample. Extract and analyze as described in Paragraph 7.	For samples that have not been treated with a decon solution that have sufficient sample volume), a matrix spike (MS)/matrix spike duplicate (MSD) pair should be analyzed. Prepare them in the same manner as the method blank spike (MBS)/ method blank spike duplicate (MBSD), but use a field sample rather than a brine solution. Extract and analyze the sample as described in Paragraph 7.	Add 0.15 mL of spiking solution to 30 mL of brine solution. [The final concentration should be at least 5 times the method detection level (MDL)]. Extract and analyze as described in Paragraph 7.
Soil or solid	Use 10 g of a representative, analyte-free material (such as DPG soil). Extract and analyze the sample as described in Paragraph 7	Add 1.0 mL of spiking solution to 10 g of sample material. Extract and analyze the sample as described in Paragraph 7	Add 1.0 mL of spiking solution to 10 g of representative, analyte-free material (such as DPG soil)[1]. Extract and analyze the sample as described in Paragraph 7

6.3 Documenting Standards and Laboratory QC Samples

This section provides procedures for technical personnel to document the prepared standards and laboratory QC samples.

6.3.1 Documenting Standards

To document all standards, record the following information about standard and spiking solution preparation in the analyst's notebook or LIMS at the time the solutions are prepared:

- Material source and lot number
- Mass or volume taken
- Final volume
- Solvent type and lot number
- Analyst's initials
- Date prepared
- Expiration date

Record all manipulations to ensure traceability from bench records to neat agent. Each preparation is identified uniquely by using the next available identity number in the Dilute Chemical Agent Logbook (an electronic spreadsheet or database tracking system may be used). Record the dates and amounts of each solution used in the logbook or electronically in the LIMS.

6.3.2 Documenting Laboratory QC Samples

To document laboratory QC samples, record the following information in the logbook or electronically in the LIMS:

- Spiking solution ID number
- Volume of spiking solution used
- Concentration of spiking solution used
- Analyst's initials
- Date prepared

7.0 Procedure

To analyze liquid or soil/solid samples by GC, analysts will perform the following tasks:

- Extract samples
- Set up the instrument
- Establish calibration
- Analyze samples using GC/FPD or GC/MSD
- If necessary, confirm FPD results using GC/MSD, or a GC/FPD equipped with a different column

7.1 Extracting Samples

Samples will be extracted within 14 days of sampling. To extract liquid samples, analysts will perform the procedures in Paragraph 7.1.1. To extract soil samples, analysts will perform the procedures in Paragraph 7.1.2. To extract solid samples, analysts will perform the procedures in Paragraph 7.1.3. In conjunction with the extraction of each analytical batch of samples (not to exceed twenty field samples), the analyst will extract a full set of QC samples as described in Paragraph 6.2.

Multi-phase or multi-layered samples (e.g., liquid-solid or liquid-liquid) will be analyzed individually if one of the phases/layers is greater than or equal to 10% of the sample. After the phases/layers are analyzed separately, a weighted average will be reported as the result.

7.1.1 Extracting Liquid Samples

To extract liquid field samples and QC samples, the analyst will perform the following tasks:

1. Uniquely identify each GC sample vial and extraction vessel that will be used to prepare the samples.
2. For each sample and QC sample, transfer a representative 30-mL aliquot into an appropriate test tube or separatory funnel.
3. If samples are to be analyzed for GB and the sample is non-reactive (e.g., ground water), add approximately 3 g of reagent grade sodium sulfate to the sample. Shake the sample vigorously to dissolve the salt. If necessary, heat the sample to approximately 30°C to aid the dissolution of the salt. If the sample is reactive (e.g., hazardous waste), sodium sulfate does not need to be added to the sample.
4. Using a graduated pipette (or other suitable device), transfer the appropriate extraction solvent or mixture into the tube or separatory funnel and cap each tube so that it is airtight. Use the following solvents to extract the indicated chemical agents:
   - 1.50 mL of chloroform for analysis of GA, GB, GD, GF, HN1, HN3 HD and T.
   - 1.50 mL of 2-(diisopropylamino) ethanol/chloroform mixture (see Paragraph 5.2) for analysis of VX.
   - 1.50 mL of 1,2-ethanedithiol/chloroform solution (see Paragraph 5.2) for Lewisite analysis.
5. Agitate the mixture vigorously on a vortex mixer or shake it by hand for a minimum of 60 sec. Allow the chloroform to settle to the bottom and/or centrifuge the sample(s).
6. From each container, transfer an aliquot of the chloroform (bottom phase) into a borosilicate glass GC sample vial. In order to prevent any further decontamination of potential agent in the extract, make sure that the aliquot does not have any (or minimal) residual sample. Cap and seal each vial. Ensure that the caps are airtight to minimize solvent evaporation.
7. Analyze the extracts within 7 days of extraction.

NOTE: The MDL and reporting limit (RL) values usually are based on a final extract volume of 1.5 mL. The final extract volume of solvent that is used in the MDL study (1.5 mL or whatever final volume is used in the MDL study) must be documented in the LIMS as the default final volume. When a sample extract has a final volume different than 1.5 mL, the MDL and RL on the report must be adjusted by a preparation factor. This preparation factor is not directly used in the calculation of sample results because the actual sample extract volume is used.

In most liquid sample extracts, including QC samples, it is appropriate to bring the final volume of the solvent in the sample extract to a volume greater than 1.5 mL (typically 5 mL) to minimize GC column degradation. If the final volume of the sample extract is increased, the amount of increase must be appropriate to support the required action limits in Table 5. The final sample extract volume then must be documented in the LIMS or logbook. The preparation factor used to correct the MDL and RL on the final report is calculated using Equation 1:

Equation 1

$$\text{Preparation Factor} = \frac{Vf}{Vm}$$

Where:

Vf is the final adjusted sample extract volume (mL)
Vm is the extract volume used for the MDL determination (mL)

Table 5: Action Levels for Waste Characterization.

Matrix	Analytical Methods[a]	Analyte[b]	Action Level	Units
Liquid	CL-002R (GC, GC/MS)	GA, GB, GD, GF, VX	0.02	mg/L
		HD, HN1, HN3, HT, Lewisite and T	0.2	mg/L
Soils/Solids	CL-002R (GC, GC/MS)	All Agents	MDL[c]	mg/kg

[a]GC – Gas Chromatography; MS – Mass Spectroscopy; DAAMS – Depot Area Air Monitoring System
[b]GA – Tabun; GB – Sarin; GD – Soman; GF – Cyclosarin; VX - o-ethyl S-(2-diisopropylaminoethyl) methylphosphonothiolate; HD - Distilled Mustard; HN1 - bis-(2-chloroethyl)ethylamine; HN3 - tris-(2-chloroethyl)amine; HT - Mustard/T; T - O-Mustard
[c]Risk-based action levels have not been determined for soils and solids. The Method Detection Limit is specific to an analytical instrument (such as GC/MS-Selected Ion Monitoring SIM, GC/Flame Ionization Detector FID, and GC/Flame Photometric Detector FPD). The MDL will be used for the action level until action levels are promulgated by Utah Division of Waste Management and Radiation Control. The Central Hazardous Waste Storage Facility may accept F999 and P999 wastes only if associated chemical agent MDL studies are up to date (see Paragraph 9.5). Soil samples are analyzed very infrequently at DPG. MDLs will be determined as needed before the analysis of soil samples. The MDLs will be maintained on file for review.

7.1.2 Extracting Soil Samples

To extract soil field samples and QC samples, the analyst will perform the following steps:

1. Uniquely identify each GC sample vial and extraction vessel that will be used to prepare the samples.
2. Mix the soil sample thoroughly so that soil removed will be as representative of the whole as possible. Mix it by shaking the closed container, stirring the contents of the container, or other effective means based on the consistency of the sample.
3. For each sample and QC sample, transfer a representative 10-g portion into a test tube or other appropriate container. Record the mass to the nearest 0.1 g.
4. For field samples add 1.0 mL of IPA, and for spiked samples, add 1.0 mL of spiking solution

let the spiking solution remain on the matrix for at least 1 minute before adding the remaining solvent; add 1.0 mL of IPA, and vortex mix the sample for 1 minute to wet the entire sample.

5. Using a graduated pipette (or other suitable device), transfer 8.0 mL of the appropriate extraction solvent into a test tube and cap each tube so that it is airtight.
6. Agitate each mixture vigorously on a vortex mixer or shake them by hand for a minimum of 60 sec. Allow the soil to settle to the bottom and/or centrifuge the sample(s) at 1,000 revolutions per minute (rpm) for about 5 minutes.
7. From each container, transfer an aliquot of the solvent into a borosilicate glass GC sample vial. Cap and seal each vial. Ensure that the caps are airtight to minimize solvent evaporation.
8. Analyze the extracts within 7 days of extraction.

7.1.3 Extracting Solid Samples

To extract solid field samples and QC samples, the analyst will perform the following steps:

1. Uniquely identify each GC sample vial and extraction vessel that will be used to prepare the samples.
2. Matrix spiking samples must be performed when solid samples are analyzed in order to measure the possible effects of non-standard matrices. For new or unusual matrices, a control sample should also be prepared that mimics the material in the actual solid samples. This is in addition to the normal MBS and MBSD samples which should be made up in the standard soil matrix to demonstrate method control.
3. For each sample and QC sample, transfer a representative 10-g portion of the solid into a test tube or other appropriate container. Record the mass to the nearest 0.1 g.

NOTE: The sample submitted to the laboratory is assumed to be representative of a specific site or area. It is important that the sub-sample analyzed by the laboratory be representative of the sample submitted. For inhomogeneous solids, estimate the weight fraction of each type of material in the sample (i.e., towels, tubing, gloves, rocks, etc.) and ensure that the 10 g portion is of similar composition.

4. For field samples, add 1.0 mL of IPA, and for spiked samples, add 1.0 mL of spiking solution. Let the spiking solution remain on the matrix at least 1 minute before adding the remaining solvent; add 1.0 mL of IPA, and then vortex mix for 1 minute to wet the entire sample.
5. Using a graduated pipette (or other suitable device), transfer 8.0 mL of the appropriate extraction solvent or mixture into a test tube and cap each tube so that it is airtight.
6. Agitate each mixture vigorously on a vortex mixer or shake them by hand for a minimum of 60 sec. Allow the solid and the solvent to separate or centrifuge the sample(s) at 1,000 rpm for about 5 minutes.
7. From each container, transfer an aliquot of the solvent into a borosilicate glass GC sample vial. Cap and seal each vial. Ensure that the caps are airtight to minimize solvent evaporation.
8. Analyze the extracts within 7 days of extraction.

7.1.4 Extracting Multi-phase Samples

Multi-phase or multi-layered samples with a distinct organic solvent layer should be analyzed individually if the solvent is greater than or equal to 10% of the drum contents. The aqueous phase should be prepared as a normal liquid sample (See Paragraph 7.1.1) and the solvent phase is analyzed by bringing up to volume.

To prepare a solvent phase for analysis, the analyst takes the following steps:

1. Separate the solvent from the aqueous phase by centrifuging the sample for up to five minutes or alternatively draw a second sample at the time of sampling and separating the solvent and organic layers. Enough solvent must be present in the sample for all requested analyses (at least 10 mL, but preferably >30 mL). Centrifuging will separate the sample into multiple layers. The solvent layer may not be the bottom layer.
2. Remove the solvent layer. Care should be taken to remove only the solvent layer and not the decon solution.
3. Remove 1.0 mL of the organic phase.
4. Using the normal extraction solvent (see Paragraph 7.1.1.4) bring to a final volume of 5.0 mL.

In the event that a multi-layer sample with a solvent phase greater than 10%, both phases will be reported. As with single phase samples, multi-phase samples will not be released from the West Desert Test Center if the weighted results of the drum exceed the action level. The weighted RL and MDL must also be below the action limit. If the action level is exceeded then further decontamination will take place and the samples will be re-analyzed.

For samples that contain a solvent layer greater than 10%, a matrix spike will be performed on the solvent layer along with the normal sample analysis. Since it is likely that the solvent contains residual decon, the matrix spike may not always have recoveries that one would expect in a clean matrix. Matrix spikes recoveries may vary and should only be used to determine matrix effect. A matrix spike that is extremely low indicates that the organic layer contains decon agent and is breaking down the matrix spike solution. Matrix effect should be narrated in the case narrative. Data should not be rejected if a matrix spike has extremely low recoveries. Low recoveries indicate that the decon is fulfilling its intended purpose.

7.2 Setting Up the Instrument

To set up the instrument, the analyst will first select the FPD or MSD method. If the FPD method is selected, follow the procedures in Paragraph 7.2.1 to set up the GC/FPD. If the GC/MSD method is selected, follow the procedures in Paragraph 7.2.2 to set up the GC/MSD.

7.2.1 Setting Up the GC/FPD

To set up the GC/FPD, the analyst will perform the following steps:

1. Use Table 6 to establish operating conditions in the instrument that produce valid initial and ongoing calibrations.

Table 6: GC/FPD Instrument Setup

Parameter	Condition
Column	• DB-5 (30 m × 0.32 mm) or equivalent • Stationary phase: 5% phenyl 95% silicone gum phase (cross linked) • Film thickness: 0.25 - 0.5 μm
Carrier gas	Helium
Flow rate	Approximately 15 mL/min for megabore columns and approximately 1 mL/min for smaller columns (depends on column ID)
Detector	FPD with appropriate filter
Column temperature	50°C, hold for 2 minutes, ramp at 10°-30°C/min to 200°C, and hold for 3 minutes
Injector temperature	200°C
Detector temperature	250°C
Sample size	1-2 μL splitless mode, 1-5 μL split mode

2. Ensure that peak shape, sensitivity, and resolution are adequate. Peaks should be symmetrically shaped with minimal tailing. Check sensitivity and resolution by injecting standard solutions during the set-up process. Peaks in the standard solutions should be baseline resolved. Sensitivity must be such that the low standard of the initial calibration is distinguishable from baseline noise. It may be necessary to adjust the detector gasses and/or the injection volume to optimize sensitivity. If adjustments fail to provide acceptable sensitivity, it may be necessary to clean the injector, trim the front end of the GC column, or change the column.
3. Obtain the baseline resolution for the analytes. It may be necessary to change the flow rate, head pressure, and/or the temperature ramp to optimize the chromatography. It may also be necessary to clean the injector, trim the front end of the GC column, or change the column.
4. Once the detector, chromatography, and injection conditions are set, maintain them for the duration of the analytical sequence. Include conditions of actual runs on the instrumental printouts.

7.2.2 Setting Up the GC/MSD

To set up the instrument, the analyst will perform the following steps:

1. Use Table 7 to establish operating conditions in the instrument that produce valid initial and ongoing calibrations.

Table 7: GC/MSD Instrument Setup

Parameter	Condition
Column	• HP-5MS (30 m × 0.25 mm) or equivalent • Stationary phase: 5% phenyl 95% silicone gum phase (cross linked) • Film thickness: 0.25 - 0.5 μm
Carrier gas	Helium
Flow rate	~1.5 mL/min (depends on column identification)
Detector	MSD in selected-ion or full-scan mode
Column temperature	50°C, hold for 2 minutes, ramp at 10°-30°C/min to 200°C, and hold for

	3 minutes
Injector temperature	200°C
Detector temperature	280°C
Sample size	1-2 μL splitless mode, 1-5 μL split mode

2. If the mass axis needs to be recalibrated, tune the mass spectrometer (MS) to perfluorotributylamine using the automatic tuning feature in the instrument software to verify the relative peak abundance and the mass axis calibration. Place a copy of the printout from this tuning session into the final data package. Verify the mass axis calibration daily when samples are being analyzed using this method. Adhere to the ion abundance limits set by the manufacturer.
3. Ensure that peak shape, sensitivity, and resolution are adequate. Peaks should be symmetrically shaped with minimal tailing. Check sensitivity and resolution by injecting standard solutions during the set-up process. Peaks in the standard solutions should be baseline resolved. Sensitivity must be such that the low standard of the initial calibration is distinguishable from baseline noise. It may be necessary to adjust the gain and/or the injection volume to optimize sensitivity. If adjustments fail to provide acceptable sensitivity, it may be necessary to clean the injector, trim the front end of the GC column, or change the column.
4. Obtain the baseline resolution for the analytes. It may be necessary to change the flow rate, head pressure, and/or the temperature ramp to optimize the chromatography. It may also be necessary to clean the injector, trim the front end of the GC column, or change the column.
5. If the GC/MSD is to be operated in SIM mode, the software must be set to monitor the correct ions (i.e., GA, GB, GD, GF, HD, HN1, HN3, Lewisite, T, and VX) at the correct times. While other ions may be monitored, these represent the minimum requirement. The software may be set to monitor all of these ions through the entire sample run or it may be set to monitor the ions for each target analyte in the retention-time window for that analyte. If the latter option is chosen, the time windows must be sufficiently wide (e.g., at least 30 sec) so that small shifts in retention times that occur with normal samples will not cause target analytes to be missed. A useful technique for the identification of false positives is to include additional ions that are not present in the normal compound that may be present in contaminants that mimic the target compound.
6. Once the detector, chromatography, and injection conditions are set, maintain them for the duration of the analytical sequence. Include conditions of actual runs on the instrumental printouts.

7.3 Establishing Calibration

To establish a calibration, the analyst generally will analyze a set of calibration standards IAW Paragraph 7.3.1. As an option, the analyst may choose to verify an existing initial calibration as described in Paragraph 7.3.2. If verifying an initial calibration fails, the analyst will establish a new initial calibration.

7.3.1 Establishing a New Initial Calibration

To establish a new initial calibration, the analyst will perform the following steps:

1. The chromatographic system can be calibrated using the internal- or external-standard technique. If the internal standard is to be used, spike the samples, standards, and extracts

with the internal standard. All standards, samples, and QC samples should be spiked with the same mass of the internal-standard solution. Internal standards are compounds that are similar in analytical behavior to the compounds of interest and not expected to be found in the samples. Analyze the initial-calibration standards in order from high to low concentration.

2. Obtain a printout of the calibration.
3. If necessary, update the placement of the retention time windows used by the software to identify target compounds using the retention times of the analytes in the initial-calibration standards.
4. Analyze the initial-calibration verification (ICV) standard.
5. Tabulate the initial-calibration standard responses and plot the initial calibration curve IAW Table 8.
6. Compare the calibration printout to the relative retention times listed in Table 9.
7. Ensure that the software is correctly labeling the peaks.
8. Verify that the ICV meets criteria (see Paragraph 8.2).

Table 8: Initial Calibration Requirements

Analysis Method	**Chemical Agents**	**Equation**	**Instructions**
FPD	GA, GB, GD, GF, and VX HD	Linear evaluation Power function or quadratic	Ensure that the correlation coefficient squared (r^2) is $\geq$0.995
MSD	GA, GB, GD, GF, VX, HN1, HN3, Lewisite, and HD	Linear evaluation, average response or calibration factor	Ensure that r^2 is $\geq$0.995. Alternatively, if the relative standard deviation (RSD) of the calibration or response factors is $\leq$15% over the calibration range, then linearity through the origin may be assumed, and the average calibration or response factor may be used to determine sample concentrations.

Table 9: Relative Retention Times and Primary Masses

Chemical Agent	**Relative Retention Time to VX (Minutes)**	**Primary Mass (MSD Analysis)**
GB	0.19	99
GD	0.39	99
HD	0.54	109
GA	0.48	70
GF	0.56	99
HN1	0.60	120
HN3	0.76	154

Lewisite	0.87	167
T	1.31	123
VX	1.00	114

7.3.2 Verifying an Existing Initial Calibration

To verify an existing initial calibration, the analyst will perform the following steps:

1. Analyze a calibration check (CC) standard.
2. Perform a QC check IAW Paragraph 8.2 on the CC analysis results. If the results are acceptable the previous, initial calibration is valid and may be used to quantitate the samples. If the CC results are not acceptable, perform the steps in Paragraph 7.3.1 to establish a new initial calibration.

7.4 Analyzing Samples Using GC/FPD or GC/MSD

To analyze samples, the analyst will perform the following steps:

1. Analyze the MB, MBS, MBSD, matrix spike, matrix spike duplicate, samples, and CC standards as shown in Figure 2. Maintain a consistent injection volume for all samples, standards, and QC samples. If matrix spike and matrix spike duplicate samples are to be analyzed, analyze them as field samples.
2. Analyze a CC standard after every ten injections of samples, blanks, and/or QC samples. The CC may be analyzed in duplicate. Samples must be bracketed by a passing CC. If CC and duplicate CC fail then those samples not bracketed by a valid CC must be re-analyzed. Further extraction and analysis may indicate that the sample is causing the chromatographic system to become overly reactive. Further corrective action or narration may be necessary. If a CC standard fails to meet the QC criteria, it may be rerun within 12 hours provided that instrument conditions have not changed and no further samples or blanks have been run. If this second analysis meets the criteria, then the analyses preceding the CC are acceptable. If both analyses of the CC fail QC criteria, then all analyses since the last passing CC must be repeated. Note that if the CC fails high for a particular analyte and that analyte is not detected in the sample, the non-detected value may be reported.
3. Ensure that QC requirements are met for each type of sample or standard (see Paragraph 8.2).
4. Clearly document QC exceptions or other changes to the method that could be considered departures from the approved method. Any changes to the method must be approved as stated in the QAPP.

7.5 Confirming FPD Results using GC/MSD

Confirmation analyses are required if peaks are observed in the retention time windows for the target analytes when using the FPD. If samples are initially analyzed by MSD, confirmation analysis may be unnecessary if the ion abundances do not meet the established ratios. If the ion abundances are similar to the ratios from the calibration standards, then further analysis may be necessary. Since the possibility exists that the positive hit is from an interferent, it may be useful to verify the hit through a full scan analysis. Use the procedures in Paragraph 7.5.1 to perform confirmation analysis by MSD. Use the procedures in Paragraph 7.5.2 to perform confirmation analysis by FPD.

7.5.1 Performing Confirmation Analysis Using MSD

Confirmation analysis using MSD may be performed in SIM or full-scan mode. To perform confirmation analysis using MSD, the analyst will perform the following steps:

1. Use a mid-range standard or spiked blank as a spectral and retention-time standard to verify instrument sensitivity (if available use a standard close in concentration to the found amount in question).
2. Analyze samples.
3. Compare sample spectra with standard spectra and/or a search of library spectra.

7.5.2 Performing Confirmation Analysis Using FPD

To perform confirmation analysis using an FPD, the analyst will perform the following steps:

1. Use a GC column that is dissimilar to the column used in the initial analysis. Use a column that causes a dramatic change in relative retention time of the target analytes, preferably inverting the elution order of at least some analytes, while maintaining acceptable chromatographic performance. Only use this type of confirmation for samples that do not produce highly convoluted chromatograms.
2. Verify the calibration using initial calibration verification and CC standards IAW Paragraph 7.3.
3. Analyze the MB, MBS, MBSD, matrix spike, matrix spike duplicate, samples, and CC standards as shown in Figure 2. Maintain a consistent injection volume for all samples. Analyze a CC standard after every ten injections of samples, blanks, and/or QC samples.
4. QC results must demonstrate that sensitivity and selectivity are adequate for positive peak identification.
5. Report results as detected only if analytes are detected in the initial analysis and are confirmed as detected in the confirmation analysis.

8.0 Data Reduction and Assessment

This section presents the following procedures performed by the Analyst to reduce data and assess QC sample results:

- Performing data reduction
- Assessing QC data
- Implementing corrective action

8.1 Performing Data Reduction

To perform data reduction, the analyst will complete the following tasks:

- Determine the validity of peaks
- Evaluate suspect peaks
- Verify RL
- Reduce the data

8.1.1 Determining Validity of Peaks

To determine the validity of peaks, the analyst will perform the following steps:

1. Consider any peak that appears in the retention time window a tentatively identified target analyte.
2. Evaluate the GC software peak selection, which is based on 5% of the retention time of known peaks in the daily standard, by using an absolute retention time window of ±0.1 min (6 s). If the peak is within this absolute retention time window, it is acceptable. If the peak is not within the retention time window, it is normally rejected; however, it may be accepted based on the judgment of an experienced analyst. If a peak outside the retention time window is accepted based on an analyst's judgment, the reasons for acceptance must be documented in the analyst's notebook and case narrative for subsequent supervisory and QA review. Inexperienced analysts or technicians should consult an experienced chemist, supervisor, or QC officer before exercising this judgment.
3. Indicate a rejection by crossing the analyte off the quantitation report and initialing and dating the cross out. A short explanation should be given for the cross out in the raw data records.
4. When evaluating analyses performed using SIM GC/MSD, evaluate the ions ratios that were acquired using Table 10, which lists the expected ions ratios found in the selected-ion scan acquisitions of different chemical agents. Use professional judgment to interpret mass spectra and the original chromatograms. Use the ranges listed in Table 10 as guidance for the target compound spectra, not as absolute acceptance ranges.
5. Ensure that the peaks from FPD analyses are 3 to 5 times the height of the noise level of the chromatographic baseline.

Table 10: Expected Ion Ratios[a]

Chemical Agent	Mass	Ion Abundance Criteria (%)
GA	70	Base peak, 100
	106	8-35
	133	20-60
	162	10-50
GB	99	Base peak, 100
	81	5-425
	125	15-45
GD	99	Base peak, 100
	69	10-50
	82	20-65
	126	70-110
GF	99	Base peak, 100
	54	5-30
	67	10-35
	81	3-30
HD	109	Base peak, 100
	63	25-55
	111	25-55
	158	8-35

Table 10: Expected Ion Ratios[a]

Chemical Agent	Mass	Ion Abundance Criteria (%)
HN1[b]	120	Base peak, 100
	122	24-35
	92	11-17
	134	D-10
	154	D-10
	85	D-5
HN3	154	Base peak, 100
	156	24-86
	63	10-61
	92	4-20
	55	1-10
Lewisite	167	Base peak, 100
	228	20-50
	165	8-35
	230	5-30
T[c]	123	Base Peak, 100
	122	33-50
	125	36-53
	124	23-34
	199	D-10
	226	D-10
VX	114	Base peak, 100
	72	20-50
	79	8-35
	127	5-30

[a]Data displayed in this table was generated from actual spectra in several analytical runs. The standard deviation was calculated and windows were defined at the 99% confidence limit plus a small additional amount in order to weight toward positive identification. Actual ratios should be compared to the daily calibration standard. Lesser Ions will have a greater variability.

[b]Data based upon a ±20% window from a single analysis performed on April 4, 2006.

[c]D indicates detect. D-5 means an ion abundance between detect and 5% of the reference ion. D-10 means an ion abundance between detect and 10% of the reference ion.

8.1.2 Evaluating Suspect Peaks

To evaluate suspect peaks, the analyst will consider the following techniques and document any change to GC parameters in the analyst's notebook and the case narrative.

- When samples produce highly convoluted chromatograms that are difficult to characterize as non-detects, change the GC conditions or utilize post-extraction spikes in an attempt to more fully characterize the samples.
- Try to separate interfering peaks from the target analyte by doing the following:
    - Lengthen the run time.
    - Slow the temperature ramp during analysis.

For example, changing the main temperature ramp to 5°C/min will provide more opportunity to elute the target analytes without interference. If this technique is used, it is important to follow all analytical protocols for the analysis or confirmation procedure, including analyzing all required samples and standards using the slow ramp.

- Use professional experience to implement other actions that may be appropriate to evaluate samples that do not provide acceptable results.
- Samples that have been analyzed by GC/MSD using SIM rather than full-scan mode have a greater potential for producing data with false positive results. In the event that a sample has all of the correct ions, proper ion abundances with the correct ion ratios, and falls within the correct retention time windows, it is appropriate to run the sample in full-scan mode to determine if the compound is truly present or a false positive. The full-scan analysis is for confirmation and qualitative purposes only and is used to determine if the suspected compound is truly present or another compound with the same ions as those in the agent compound. A calibration standard will be analyzed for qualitative purposes (i.e., to determine the detection and retention times of the compounds) and not for any quantitative purposes. The review of these full-scan samples must be performed by an analyst who is familiar with the review and interpretation of GC/MS data. The sequence of analysis for the full-scan spectra is as follows:
    1. Perform a perfluorotributylamine tune as described in Paragraph 7.2.2. This is done to verify mass axis calibration and correct ion ratios used in scan analysis.
    2. Analyze a low to mid-range calibration standard of the suspected compound. Use the same oven program that was used in the SIM analysis. This will help to confirm the retention time of the suspected compound and to provide the mass spectra of the suspect compound. The only requirement for this standard is that the compound be present and identifiable.
    3. Analyze one or more reagent blank(s) to verify that the analytical system is free of contamination.
    4. Analyze sample.
    5. Compare sample spectra with the spectra from the calibration standard in Section 8.1.2 Paragraph 2.
    6. Perform library search of the compound of interest to see if it matches any of the entries in the National Institute of Standards and Technology (NIST) library.
    7. The determination of the presence or non-presence of the compound should be carefully studied. The resulting spectra as well as the contents of the sample or waste stream should be evaluated. For example if a sample is taken from a source with a high pH or an environment in which it is impossible for the compound to exist this should be evaluated along with the GC/MS data.
    8. Narrate in the analyst log book and/or in the case narrative the results of such findings.

8.1.3 Verifying RLs

If a sample produces a highly convoluted baseline, it may be necessary to verify that the RLs are achievable. To verify RLs, the analyst will perform the following tasks and document them in the analyst's notebook and case narrative.

- Use a post-extraction spike to demonstrate whether or not the target analytes can be detected at the RL. To use this option, spike an amount of the chemical agent spiking solution into a

measured aliquot of sample extract so that the final concentration of analyte in the extract is at approximately the RL. For example, 100 µL of a 4.0-µg/mL spiking solution spiked into 1.0 mL of extract will yield target analytes at approximately 0.40 µg/mL.

- Analyze the spiked extract under the same conditions as the unspiked field samples and attempt to identify the chemical agent in the spiked sample.
- Report the non-detected result for the field sample if the chemical agent is identifiable in the spiked extract but not in the unspiked extract.
- Raise the MDLs of the analysis if the chemical agent is not identifiable in the spiked extract.
- Perform additional spikes at higher concentrations to estimate the actual MDLs for a particular sample if it is necessary for the data user.

8.1.4 Reducing Data

To reduce data, the analyst will perform the following tasks:

- Determine the extract concentration by comparing the instrument response for a sample with the equation for the initial calibration curve. This comparison may be performed manually or using the GC software or integrator.
- Use the batch spreadsheet to calculate sample concentrations and spike recoveries. Use Equation 2 to calculate the final concentration when dilutions and/or extractions have been used.
- Elevate RLs by any dilution factors included in the analysis.

Equation 2

$$\text{Reporting Concentration} = \frac{Q \times df \times Vf}{E} \times U$$

Where:

Q is the concentration determined by comparison to the calibration curve (typically mg/L, µg/L, or mg/kg).
df is the final dilution factor (if needed).
Vf is the final extract volume (mL).
E is the extracted sample volume (mL) or weight (g).
U is the unit conversion factor, such as µg to mg (if needed).

8.2 Assessing QC Data

To assess QC data, technical personnel will ensure that the QC samples listed in Table 11 are analyzed. For the sample results to be considered acceptable, the results must meet the criteria in Table 11. Possible corrective actions for QC failure are also listed in Table 11. Other corrective actions may be considered based on the experience of the analyst. Document all corrective actions in the analyst's notebook.

Table 11: Quality Control Criteria

Quality Control (QC) Sample	Equation	Criteria	Corrective Action
Initial Calibration	First- or second-order regression	(r^2) is ≥0.995	Recalibrate the instrument and rerun all the samples.
Initial Calibration Verification	*Equation 3* $\%R = \frac{Found}{Expected} \times 100$ Where: Found is the sample result Expected is the standard concentration	% R 85 to 115%	Recalibrate the instrument and rerun all the samples.
Calibration Check		% R 80 to 120%. Every sample must be bracketed by valid CC standards or initial calibration. If the calibration fails high (i.e., high bias), then the closing continuing calibration may be used provided that the failed analyte is not detected in the samples (see Section 7.4 Paragraph 2).	Rerun all the samples before and after the failed CC standard, to the next valid CC standard. Initiate corrective action.
Cleanliness	NA[2]	MB concentrations must be < RL.	Initiate corrective action.
Accuracy	*Equation 4:* $\%R = \frac{SSR - SR}{SA} \times 100$ Where: SSR is the spiked sample result SR is the unspiked sample result SA is the spiked amount	% R must be between 60% and 140%, inclusive.	Initiate corrective action.
Precision	*Equation 5:* $\text{RPD} = \frac{2\lvert MBSR - MBSDR \rvert}{MBSR + MBSDR} \text{ X100}$ Where: MBSR is the MBS percent recovery MBSDR is the MBSD percent recovery	RPD must be ≤25%.	Initiate corrective action.

[1]Recovery.
[2]Not applicable.

8.3 Implementing Corrective Action

If QC parameters do not meet the requirements listed in Table 11, the Analyst shall implement the following corrective actions:

- For initial calibrations, reanalyze the initial calibration and any samples that have been analyzed using this curve. Possible corrective actions for a calibration failure include injection port maintenance, column maintenance, and re-preparing standards.
- For CCs if no adjustments have been made to the instrument and no further samples or blanks have been analyzed since the failing CC, the CC may be re-injected within 12 hours. If the second injection passes, the re-injected value is acceptable as a CC. If the second injection also fails, reanalyze all of the samples injected since the last passing CC on a compliant system. Note that if the CC fails high for a particular analyte and that analyte is not detected in the sample, the non-detected sample may be reported (see Section 7.4 Paragraph 2). Possible corrective actions for CC failure include injection port maintenance, column maintenance, and re-preparing standards.
- For MBs, inspect the GC for possible sources of carry-over. Cleaning the injection port may solve this problem. If the contamination cannot be traced to the instrument and is in the blank extract, re-extract and reanalyze all of the samples extracted with the MB. If the blank shows contamination but the samples show no positive peaks and all other QC parameters are within limits, the samples may be reported with noted exceptions.
- For MBSs, the recoveries (accuracy) for these samples should be between 60% and 140%, inclusive, and the RPD between them (precision) should be $\leq$25%. If the results are outside the limits, reanalyze all of the associated samples. Appropriate corrective action may include injection port maintenance and column maintenance. If upon reanalysis the recoveries are still outside of the criteria in Table 11, re-extract and re-analyze all of the associated samples. If the spike recoveries are high and above the upper limit, and the sample results are non-detected, the results may be reported with noted exceptions.
- For matrix spike and matrix spike duplicate, the recoveries (accuracy) for these samples should be between 60% and 140%, inclusive, and the RPD between them (precision) should be $\leq$25% If the results are outside the limits, but the results for the MBS and MBSD are within limits, the recovery problem is considered to be matrix-related. The client should be notified that the recoveries from the matrix are suspect. If the sample was at a pH that indicates the presence of decontamination solutions, this should also be noted to the client.
- For ICVs, reanalyze the ICV. If the results are still outside criteria, recalibrate the instrument and reanalyze any samples that have been analyzed using this curve. Possible corrective actions for a calibration failure include injection port maintenance, column maintenance, and re-preparing initial calibration or verification standards.

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

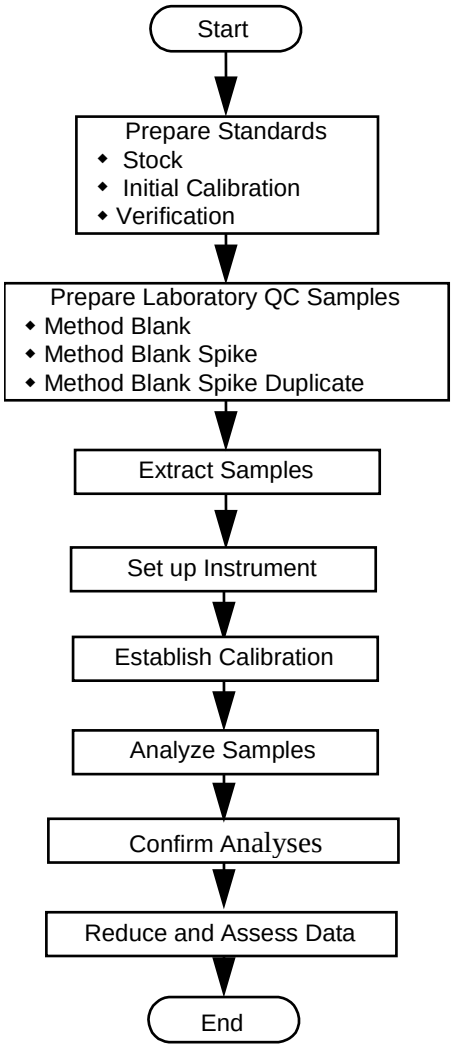

Figure 2. Typical Analytical Sequence