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STATEMENT OF WORK (FORMERLY CALLED "REQUIREMENTS DOCUMENT") - ANALYTICAL SUPPORT
(RFP number 9-XS1-Q4257)
(REVISION 2 - JULY, 1995)

I. BACKGROUND

I.A. Overview of the problem - The University of California has operated Los Alamos National Laboratory since 1943. The primary mission of LANL has been nuclear weapons research and development and basic research in the areas of physics, chemistry, and engineering that supports the weapons development. In addition, research on peacetime use of nuclear energy has included space applications, power reactor programs, radiobiology, and medicine. Other LANL programs include elementary particle physics, applied photochemistry, astrophysics, earth sciences, energy resources, nuclear fuel safeguards, lasers, computer sciences, solar energy, geothermal energy, biomedical and environmental research, and nuclear waste management research. Past and present work performed at LANL has created a need for analyses of samples, both for the purpose of Waste Management activities related to treatment, storage, and disposal of wastes generated as a result of ongoing operations (radioactive, hazardous, and mixed waste), and for the purpose of Environmental Restoration, which includes remedial actions involving past and potential releases from inactive waste sites, and decontamination and decommissioning of surplus facilities. Sample analyses are needed primarily in the areas of organics, inorganics, radiochemistry, and high explosives.

LANL must comply with the Atomic Energy Act and all Federal and State environmental requirements addressing the handling, transport, release, and disposal of hazardous materials, as well as protection of ecological, archaeological, historic, atmospheric, and aquatic resources. The primary Federal legislation impacting the analyses of environmental samples for LANL is the Resource Conservation and Recovery Act (RCRA) as amended by the Hazardous and Solid Waste Amendments (HSWA) of 1984. The Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) and Superfund Amendments and Reauthorization Act (SARA-related actions for potential release sites are being addressed under DOE's Environmental Restoration Program in conjunction with RCRA corrective actions. Other environmental requirements, such as the Clean Air Act, Clean Water Act, Safe Drinking Water Act, and State requirements may influence analytical needs, such as method detection and quantitation limits, analytes of interest, and media of interest.

I.B. Purpose of subcontracts - Presently, LANL internal laboratories provide chemical analytical services for the Laboratory, including organic, inorganic, and radiochemical analyses for trace level contaminants in various matrices, such as water, waste water, soils, sludges, filters, and oils. These matrices may contain both radioactive and hazardous materials.

LANL also uses subcontractor support for analyses of samples (through the subcontracts awarded under RFP number 9-XS1-Q4257) because of the workload related to RCRA and other activities in the areas of Waste Management and Environmental Restoration, at a minimum, that exceed the capacity present within LANL internal laboratories. The subcontracts provide LANL with support in the areas of organics, inorganics, radiochemistry, and high explosives. Samples may contain both hazardous and radioactive components. Radioactivity will not exceed 100 nanocuries (nCi) per gram.

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Procedures for both LANL and the subcontractors follow, as well as general and specific subcontract requirements as revised through Revision 2.

II. GENERAL REQUIREMENTS

II.A. Sample scheduling and receipt procedures - The responsibility for scheduling of samples rests with LANL's Sample Coordinator. The Sample Coordinator will make every effort to notify the subcontractor of sample delivery at least 4 weeks prior to the dispatch of samples from LANL to enable the subcontractor to effectively schedule sample analyses. When the subcontractor has been notified at least 4 weeks in advance of sample delivery (assumed to be one business day - including Saturdays - after the day of dispatch), it is obligatory to accept the samples and analyze them according to specified requirements (i.e., perform the required analyses within holding times and deliver the data by the due date). In the case that the subcontractor is notified of sample delivery less than 4 weeks prior to dispatch of samples, the subcontractor has the right of refusal. In extenuating circumstances, when the subcontractor has had at least 4 weeks notice of sample delivery and will be unable to meet the data turnaround time, the Sample Coordinator will consider negotiating (with the approval of the Contract Administrator) an extended data delivery date with commensurate sample analyses price reductions, providing this negotiation occurs prior to sample delivery.

If sample delivery dates change by more than 5 days due to field problems or client sample scheduling changes, unless the laboratory is notified of these changes at least 4 weeks prior to shipment of the samples, the subcontractor's right of refusal is in effect.

Scheduling of sample delivery may be by telephone and/or FAX during the initial phase of the subcontract. However, it will be done electronically by the Sample Coordinator as soon as possible. In addition, electronic transmission of the analysis request will be coordinated with sample delivery, identifying, at a minimum, the Sample Delivery Group (SDG) or Request Number (RN) ("SDG" and "RN" will be used interchangeably to mean the same thing - based on the specific LANL program's usage), sample numbers, analyses to perform, and required turnaround time. The subcontractor must notify the Sample Coordinator, either by phone or FAX (may be electronically, in the future), if the samples are not received on the day scheduled. In addition, when samples are received, the subcontractor must notify the Sample Coordinator of any problems associated with those samples (e.g., broken containers, inadequate volume - not problems with the analyses) by the next business day.

Whatever information can be provided about the radioactivity of the samples being scheduled will be communicated to the subcontractor by the Sample Coordinator. The results of LANL's radioactivity screening may preclude delivery of scheduled samples if higher radioactivity is identified then allowed by the laboratory's Nuclear Regulatory Commision (NRC)/agreement state license. Any screening data provided with the samples is not guaranteed to be accurate enough to preclude the need for the laboratory to perform whatever radioactivity screening it considers necessary to protect health and safety and NRC licenses.

LANL plans to supply all sample bottles and coolers for this subcontract. However, in an emergency, the subcontractor may be asked to supply bottles and/or coolers, which will be billed as a separate line item according to the subcontractor laboratory's standard price list for this service, which will be submitted with your best and final offer. Specifications for the bottles/coolers will be provided by the Sample Coordinator if the need arises.

Sample delivery will be by SDG or RN. An SDG or RN is defined as a group of samples that must be reported together (may include QC samples), and may arrive at the subcontractor laboratory over a period of up to 14 days, to allow for long-term field sampling when necessary. Note that the SDG or RN generally arrive all in the same day. The subcontractor must meet holding times, regardless of the fact that an SDG/RN may be delivered to the laboratory over up to a 14 day period. The data due date for the entire SDG/RN will be based on the last day of delivery of samples for that SDG/RN. The Sample Coordinator will designate the sample numbers that fall within an SDG or RN, based on the sample matrices that are to be analyzed.

It is important to understand how LANL is defining a sample for purposes of this subcontract and how it relates to the SDG or RN (though not related to sample analyses prices). The Sample Coordinator or field personnel will assign a sample number to each sample bottle delivered to the subcontractor, with the appropriate designation of determinations to be performed on the template accompanying the samples. The SDG or RN count is based on the number of different sample numbers received by the subcontractor laboratory. There may be more than one "determination category" for a sample - e.g., for organics there could be 3 determination categories for one sample (VOA, BNA, and pesticide). There could be several bottles delivered for the single sample for VOA, BNA, and pesticide analyses, but all would have the same sample number on them, and they would count as one sample of the SDG.

LANL reserves the right to order analyses for specific analytes under these "determination categories" (such as a single metal or a limited list of VOAs) for which the subcontractor would report only the results requested but can be paid as if the entire determination category was ordered. The reason for this is for ease of data review/data assessment when there are specific contaminants of concern identified by LANL. The rationale for payment as if the entire determination category were reported is that the subcontractors are likely set up in a mode to routinely produce data on the gamut of analytes within a determination category (calibration, reporting forms, etc.) so that they do not save money by analyzing a limited list from the determination category. Not withstanding any of the above paragraph, the subcontractor may elect to charge a lesser amount when a limited list of analytes is ordered - as may be proposed under non-routine analytical services.

All samples dispatched from LANL that may be from an area of possible radioactive contamination will undergo radiological screening with the exception of rinsates and some water samples. (The subcontractor will be paid the % increase in sample analysis cost for radioactive samples that is agreed upon at subcontract award.) When available, this screening data will be provided to the subcontractor. (Procedures for radiological screening are found in Appendix 1.) No samples will knowingly be sent to a subcontractor that exceed the subcontractor's NRC/agreement state license. Note that the subcontractor is required to provide LANL with a copy of any new or revised NRC licenses upon their receipt. Note also that all laboratories receiving samples from LANL that may be from an area of possible radioactive contamination must have an NRC facilities license. If a laboratory conducts their own radiological screening and determines the sample to be at background, the sample may be sent for analysis to a "sister" laboratory that is included on the contract. In such an event LANL will not be responsible for any damages caused by inaccurate radiological screening by the subcontractor. Each laboratory is responsible for ensuring that such shipments are within the applicable NRC/agreement state regulations.

Despite the fact that LANL will screen the samples for radioactivity prior to shipment, and will not knowingly send samples to the laboratory exceeding the NRC facility license limit, it is the subcontractor's responsibility to manage any potential exceeding of the license limit due to other

samples in house. The subcontractor must work with the Sample Coordinator if it appears the license limit may be exceeded; however, once the capacity is committed to LANL, the subcontractor must not take other clients' samples if those samples will cause the laboratory's license limit to be exceeded once LANL samples arrive.

II.B. **Deliverables requirements** - Data turnaround times may be from 15-60 days for organics, inorganics, and explosives and from 30 days to a subcontractor-proposed turnaround time for radiochemistry, at the specific prices for various turnaround times established at subcontract award. The schedule for delivery of data will be provided by the Sample Coordinator at the time of scheduling.

Initially, the deliverables will consist of Microsoft Excel spreadsheets or Tab delimited ASCII files on 3½ inch diskettes, a Case Narrative in Microsoft WORD or Wordperfect (as described in Section V) as well as hardcopy for elements not transmittable in Microsoft Excel (e.g. chromatograms, spectra). In addition The required hardcopy items are identified in each of the six routine analyses Sections IIIs and must be delivered by the data due date (with the diskette, if it is provided). LANL will provide the software needed for generating the LANL-specific Microsoft Excel spreadsheets/TAB delimited ASCII files.

A hardcopy of the Case Narrative described in Section V, "Reporting Requirements," is required with delivery of data for each SDG/RN.

A copy of all laboratory Chain-of-Custody documentation must also be delivered simultaneously. Note that the original Chain-of-Custody documentation will be transmitted with the samples returned to LANL unless samples are completely used up or, with LANL approval, the laboratory disposes of the samples, in which case the original is sent with the diskette/hardcopy, as described in II.E.

All reports and documentation must be legible, complete, paginated, and in order according to requirements identified in the template accompanying the samples, and paginated.

Soil results must be reported on a dry weight basis (including % moisture) except for tritium and other instances when it is inappropriate to dry the soil sample prior to analysis (in which case the % moisture should be reported, as for tritium) **unless it creates a potential hazard to dry the sample to calculate the percent moisture**. In these cases, it must be made clear in the case narrative (essentially an SDG narrative) why the sample was not dried and % moisture not reported.

Examples of the draft Microsoft Excel spreadsheets to be used are located in Appendix 2.

The hardcopy and the SDG file (see Section IX.C) must be sent together (with the diskette, if requested) to:

John Miglio, Sample Coordinator
Los Alamos National Laboratory
PO Box 1663
MS E-509
Los Alamos, New Mexico 87545

or, if sent by overnight carrier such as Federal Express,

John Miglio, Sample Coordinator
Los Alamos National Laboratory
TA-35, Building 128. MS E-509
Drop 01U
Los Alamos, New Mexico 87545

The required electronic deliverable is an IBM or IBM-compatible, 3.5 inch diskette.

The hardcopy case narrative and items spelled out in the specific routine analyses categories must always be submitted. Hardcopy of the diskette deliverable will be requested when needed, and is separately billable. CLP-like forms may be requested by form number also. If this occurs, they will be billable separately.

II.C. Raw Data Delivery - Raw data is considered part of the SDG/RN File as described in Section IX, and is transmitted as part of the SDG/RN File by the data due date.

II.D. Sample holding times - All samples must be prepared and analyzed in accordance with relevant holding times from the sampling date as spelled out in SW-846. The analysis request documentation will clearly relate the sampling date/time so that the subcontractor is aware of the time constraints on sample preparation or analyses. LANL plans to have samples delivered to the laboratory with at least 72 hours remaining for sample extraction or analysis. However, it may occur that the samples arrive in the laboratory with less than 72 hours remaining within the SW-846-stipulated holding time and it may be critical to the user of the data that the holding time be met. In these circumstances, the subcontractor would be paid the % sample analysis price increase established for sample extraction or analysis on samples arriving with less than 72 hours remaining toward the SW-846 holding time. The subcontractor will not be held responsible for missing holding times when LANL has sent the sample(s) too late to meet SW-846 holding times (e.g., 24 hours or less until holding time expiration).

II.E. Return of unused samples and radioactive digestates and extracts - All unused samples, unless LANL has approved disposal by the laboratory, and all empty coolers must be returned to LANL (Sample Coordinator) in the original sample containers. Samples must be returned to LANL within 60 days of LANL's acceptance of data. *The subcontractors will be provided with instructions for using a LANL billing number (also for data package delivery).* Unused samples must be returned under chain-of-custody, using the original Chain-of-Custody documentation for this purpose and must be properly packaged to prevent breakage. This chain-of-custody documentation for return of samples will be included, by LANL, as part of the SDG/RN File described in Section IX.

In addition to the unused samples, LANL will accept for return samples that were analyzed "as is" (such as samples used for gamma spectroscopy) if returned in the original container. Radioactive digestates and extracts can also be returned if the radioactivity is greater than the measured background by the technique used by the laboratory. However, LANL will not take back hazardous waste extracts and digestates unless they are radioactive, with the radioactivity greater than measured background. The subcontractor laboratory will be responsible for ensuring that only those radioactive extracts and digestates that result from LANL samples are shipped back to LANL. They must be shipped back under chain of custody and clearly labeled with sample numbers and analysis types, since LANL will need clear definition of the constituents/radioactivity of those extracts and digestates. If the subcontractor chooses to return these extracts and/or digestates (note that it is not a requirement), the Sample Coordinator should be contacted for

direction on how the extracts and digestates are to be screened for radioactivity - whether by the SDG/RN with the extracts or digestates composited, or by the individual extracts and digestates.

II.F. Location of performance of work - The subcontractor must perform all work associated with this subcontract at the subcontractor's facility that was audited and/or accepted by LANL. As the subcontractor develops other facilities to provide these services, a request can be made of LANL for approval of those facilities.

II.G. Hardware/software - Each of the analytical methods cited in the "specific determination categories" sections identifies the needed equipment (hardware, and in some cases software). The subcontractor must maintain all of the needed equipment in good working order for the period of performance of the subcontract. In addition, the subcontractor must have sufficient backup equipment to meet sample analyses commitments in case of equipment failure.

Microsoft Windows Operating System is required as well as Microsoft Excel

As mentioned in II.B., LANL will provide the file templates needed for the Microsoft Excel spreadsheets.

II.H. Performance Evaluation Sample results

The subcontractor must be aware that LANL will be obtaining information on Performance Evaluation Sample results from DOE's Integrated Performance Evaluation Program (IPEP). The subcontractor must provide permission, if requested, to allow provision of this information to LANL by laboratory name. All subcontractors must be registered with the Mixed Analyte Performance Evaluation Program (MAPEP) (unless they do not have an NRC facilities license). Note that MAPEP results will be provided to LANL through IPEP.

II.I. Payment reductions for non-adherence to requirements -

1. When the quality of the data is so poor because the subcontractor did not meet requirements because of deficient performance, and the user cannot use the data, there will be no payment for the sample analyses.

2. If the user cannot use the data for the intended purpose because the subcontractor did not meet requirements, but the data can be used for a lesser purpose by the user (e.g., focus future needed analyses and/or sampling points, provide screening level data), 50% of the agreed-upon price will be paid.

3. For late delivery of data, payment will be reduced by 2% per day up to 75%, unless a modified data due date was negotiated, as described in Section II.A.

Note: LANL will consider that the prices proposed by the subcontractor are for data delivered by the due date and according to subcontract requirements.

II.J. Invoicing instructions -Invoices will be submitted monthly. Each month's invoice will include all SDG/RNs/RNs analyzed during the period in question and for which data packages have been sent to LANL. Information that must be included on the subcontractor's invoices is:

- Invoice number

- Invoice date
- Subcontractor name
- Sample Delivery Group Number(s) and/or Request Numbers being invoiced
- List of sample numbers with associated determination categories, along with the price of each determination
- Total amount invoiced

Sample analyses prices for various combinations of turnaround times and monthly delivery of samples (capacity) up to the maximum for which you would like to be considered for delivery, will be established at subcontract award. This maximum, which should be based on your personnel, equipment, and facility limitations (by turnaround time) will be the basis for billing LANL. (This assumes that the higher the volume of analyses the lower the sample analysis price).

Note: If LANL did not commit to send samples during any month and no samples were sent, or no analyses were completed, from previously submitted samples, and data packages sent, there will be no payment for that month.

III. SPECIFIC REQUIREMENTS FOR ROUTINE ANALYSES

The analyses categories of organics (including volatiles, semivolatiles, and pesticides/Aroclors), inorganics, radio-chemistry, and high explosives are considered to be routine analyses for purposes of this subcontract, and are referred to throughout as **"routine analyses categories."** There will be methods options cited for each, such as SW-846, CLP, USATHAMA, and LANL. ***Any other methods/method modifications the subcontractor wishes to use must be approved by LANL. The request for approval must be sent to the Sample Coordinator, who will provide it to the appropriate person(s) for evaluation and approval. Note that only methods/modifications that are compatible with the EDD will be considered.*** The citations will be for the commonly used methods, some of which are considered to be standards. When the laboratory chooses to use cited SW-846 methods, the current (or most recent draft) version will be considered the appropriate one and will be cited. These SW-846 methods are available through the Methods Information Communication Exchange (MICE) at (703)821-4789. Note, It is not a requirement to use the promulgated version of SW-846 except in specific categories as outlined in 40 CFR Parts 260-270. The laboratory will be notified in the *unlikely* situation that samples are related to one of those categories.

In addition, a listing of the required hardcopy data is provided. The target analyte lists will be included in each of the Section IIIs, and is required - regardless of what method is used by the laboratory unless a limited list of analytes is ordered (as described in Section II.A. Each of the Section IIIs will clarify the QC requirements, regardless of the method used.

"Estimated Quantitation Limits (EQLs)" are provided for the organics, high explosives, and radiochemistry parameters. EQL's are defined in various SW-846 methods as "the lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is generally 5 to 10 times the Method Detection Limit (MDL). However, it may be nominally chosen within these guidelines to simplify data reporting. For many analytes the EQL is selected from the lowest non-zero standard in the calibration curve. Sample EQLs are highly matrix-dependent. The EQLs listed in SW-846 methods are provided for guidance and may not always be achievable." The definition indicates that EQLs listed for soil/sediment are based on wet weight and that normally data are reported on a dry weight basis, thus causing EQLs higher than those cited for dry weight.

For inorganics, "Estimated Detection Limits (EDLs)" are provided instead of EQLs, to reflect the Contract Required Detection Limits (CRDLs) of the Contract Laboratory Program methods.

Each of the 6 categories has a separate Section III, so that each subcontractor will have to deal only with the relevant sections. Note that the organics category is divided into separate Section IIIs for volatiles, semivolatiles, and pesticides/Aroclors.

IV. <u>SPECIFIC REQUIREMENTS FOR NON-ROUTINE ANALYSES</u> - Non-routine analyses which have been included in your contract at your request or may be included as a result of revisions/change orders to your contract (a revised list of LANL needs for which proposals are wanted is included as a revised Appendix 3), are analytical services other than the six routine analyses categories. Routine, standard, or widely used methods are available for some of them. Others are very specialized and may be of an emerging nature. A per-sample price for analyses for each of the parameters identified that you are interested in (and for which you have the capability), as well as a turnaround time for data delivery, should be proposed, and the method cited (if "standard") or general technology (if not "standard"). Submission of the Subcontractor price list for all available services is appropriate, if desired. The purpose of this activity is to provide LANL with a ready list of subcontractor analytical services to meet a diversity of LANL needs needs in circumstances when the 6 specified routine analyses categories are insufficient. **Note: proposals on the non-routine analyses will not affect the selection of the laboratories for subcontract awards, but are for use after subcontracts are in place.** These analyses will be ordered/scheduled in the same manner as described in Section II.A.

V. <u>REPORTING REQUIREMENTS</u> - For the 6 routine analyses categories, reporting must be made as described in Section II.B., "Data delivery requirements" and, more specifically, as defined within each of the routine analyses categories' Section III, deliverables. All results must be reported on a dry weight basis other than the exceptions noted in II.B.

Reporting for <u>non-routine analyses</u> (discussed in Section IV) will include analytical results with all supporting quality control documentation in an electronic format that resembles the routine analyses deliverables most like the non-routine, if possible. For analyses that may not in any way resemble the 6 routine categories (such as geo-technical or biological testing parameters), an electronic format can be agreed upon with the Sample Coordinator, though is not required. The minimum requirements for deliverables for all non-routine services (with the exception of measurements for which some or any are not applicable) are

- target analytes/measurement parameters and associated quantitation or measurement limits
- citation of sample preparation and analytical method used (when a "standard" method used or a description of the technology used when a standard method cannot be cited.
- calibration data
- raw analytical data (instrument outputs)
- manual calculation used for generating results (unless specified in cited method)
- analytical data for use
- all quality control documentation

A narrative called the "case narrative" (WORD or WORDPERFECT file on the diskette deliverable) will be required for every SDG/RN for both the routine and non-rouine analyses. In

addition, a hardcopy of the case narrative is required with the other specified hardcopy deliverables.

Required components of the case narrative follow:

- Laboratory name and subcontract number
- SDG/RN number
- Sample numbers included in the SDG/RN
- Documentation of quality control, sample, shipment and/or analytical problems in processing the samples, anomalies noted in the data, decision trees/corrective actions taken to solve problems, and any other information that might be of use to the data review personnel or users of the data in making decisions.

In addition, the case narrative must contain the following statement: "I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except as detailed in this case narrative." This statement must be immediately followed by the name of the Laboratory Manager or designee, title, and date of signature. The hardcopy of this case narrative must be signed in original and dated by the Laboratory Manager or designee over or next to the typed name, title, and date. This statement clearly makes the laboratory accountable for data package verification prior to delivery to LANL.

VI. REQUIRED QUALITY CONTROL PROCEDURES AND CRITERIA - Each of the routine analyses categories Section III documents contains a Section IV specific to the category and determinations within the category (e.g., organics - pesticides) which defines quality control (QC) procedures to follow and criteria to be met regardless of the method option used (e.g., CLP or SW-846).

In some cases, both SW-846 and CLP methods are cited as options. There are some differences in these documents (e.g., QC procedures and criteria), generally the CLP methods having more QC requirements. In a few cases, SW-846 criteria are more stringent. The Section III for each routine analytical category **stipulates the LANL QC requirements and criteria**. There are a few instances where procedures are different (e.g., SW-8081 requires a 5 point calibration, whereas CLP pesticides/Aroclors methods require a minimum of a 3 point calibration). The spreadsheet deliverable will accommodate use of either SW-846 or CLP methods.

In instances where the data acceptance criteria are not met, the subcontractor is required to repeat the sample preparation and/or analysis. If the criteria are still not met because the problem is caused by matrix effects the subcontractor should bill for both sets of analyses. If the problem is corrected by the reanalysis, the subcontractor should bill only for the analysis that meets the acceptance criteria and should not send the data for the non-acceptable analysis. If holding times have expired prior to the repeat of the sample preparation or analysis, the laboratory should contact the Sample Coordinator for a decision as to whether to perform the repeat.

VII. QUALITY ASSURANCE (QA) PROGRAM - The subcontractor must maintain a QA Program that ensures the quality of the data generated by the subcontractor meets the specifications of the client. There are many elements of a QA program that need to be considered such as internal QC samples to check on instrument and operator performance, use of control charts to identify warning signals before they become problems (e.g., for instrument performance, surrogate recoveries, laboratory blank contamination, standards degradation, etc.), operator training - including "refresher training," internal certification of operators, internal auditing of laboratory

operations (e.g., adherence to Standard Operating Procedures and update of them when appropriate), audits by an outside party, use of NIST and EPA reference materials to provide traceability to national standards, insertion of blind QC samples and other indicators to ensure that the laboratory identifies and corrects analytical problems as soon as possible, and verification procedures to ensure that all corrective action loops are closed. Good laboratory practices must be adhered to at all times.

The subcontractor must have a Quality Management Plan (QMP), formerly referred to a Quality Assurance Program Plan (QAPP), that meets the DOE requirements of DOE Order 5700.6c, where appropriate, and "EPA Requirements for Quality Management Plans - EPA QA/R-2 Interim Final, August 1994 (or final when available) which supersedes QAMS-005/80. The ANSI/ASQC E4-1994 "American National Standard Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs" provides minimum specifications and guidelines that apply to common or routine quality management functions and activities necessary to support environmental programs and specification and guidelines that apply to project-specific activities involving the generation, collection, analysis, evaluation, and reporting of environmental data. Applicable standards and guides that can be used to define criteria related specifically to analytical laboratories include ASTM E994, *Criteria for Assessing Laboratory Competence*, ISO Guide 25, *General Requirements for the Competence of Calibration and Testing Laboratories*, and ANSI/ASQC Q2-1991, *Quality Management and Quality System Elements for Laboratories - Guidelines*. The QMP must be available for review upon site visits or upon request and delivered to the Sample Coordinator.

LANL's QA oversight program for the subcontracts may include blind QC samples, periodic on-site visits, and data package audits as well as tracking and trending of all performance information. The subcontractor will be provided with reports on performance and on-site visit findings and corrective action items. A corrective action program will be in place requiring subcontractor response to observed and reported problems.

VIII. <u>DATA MANAGEMENT REQUIREMENTS</u> - With the ever-increasing reliance of analytical laboratories on automation in all phases of laboratory operations, it has become more and more critical to recognize the importance of good automated laboratory practices in order to ensure that the benefits of these systems are not destroyed by errors introduced as a result of use of these systems. In order to protect the integrity of computer-resident data, there is a series of minimum practices and procedures that the laboratory must follow in order to assure the data are of sufficient integrity to be used in making decisions involving human health and environmental protection.

Because of concern about computer data integrity, the Environmental Protection Agency has published and widely distributed a document entitled "Good Automated Laboratory Practices" which will, henceforth, be referred to as GALP. (Note: This document is still in draft form.) Despite the draft status, this document is considered to be an appropriate guidance document. There are 14 areas noted in the GALP for which the minimum practices and procedures must be adhered to by the subcontractors. Those areas are as follow:

- Personnel
- Laboratory Management
- Responsible Person for the automated data collection system
- A Quality Assurance Unit
- Facilities

- Equipment
- Security
- Standard Operating Procedures
- Software
- Data Entry
- Raw Data
- Records and Archives
- Reporting
- Comprehensive Ongoing Testing

While the GALP provides options for implementing minimum good automated laboratory practices, each laboratory is free to choose the approach most appropriate and effective for the organization. The subcontractors will be expected to be able to demonstrate their data management practices that respond to the objectives (called "explanations") defined within each of the 14 cited areas.

There are 6 principles that provide the foundation for good automated laboratory practices in order to provide control of the laboratories' automated systems. These principles follow:

- The system must provide a method of assuring the integrity of all entered data.
- The formulas and decision algorithms employed by the system must be accurate and appropriate.
- An audit trail that tracks data entry and modification to the responsible individual is critical to the control process and must be implemented.
- A consistent and appropriate change control procedure capable of tracking the system operation and application software is also critical to the control process and must be implemented.
- Appropriate user procedures must be followed to maintain the control process. There must be clear directions and Standard Operating Procedures (SOPs) developed; all users must be trained; appropriate user support documentation must be provided.
- In order to maintain consistent control of the system, alternative plans for system failure, disaster recovery, and unauthorized access must be developed.

Each of these six principles is a specific requirement of this subcontract, and failure to be able to demonstrate that each of them has been met could result in sanctions after award, until the provisions have been met.

IX. PROCEDURES FOR CHAIN-OF-CUSTODY RECORDS, DOCUMENT CONTROL, SDG/RN FILE PREPARATION, AND CONFIDENTIAL INFORMATION

IX.A. Chain-of-Custody - The subcontractor must have a program in place to maintain the COC record for each sample delivered to the laboratory. This COC record begins in the field when the sample is collected and ends only when the sample is returned to LANL (Sample Coordinator) or it is finally disposed of. The purpose of developing a COC record is to create an accurate written (or electronic) record that can be used to trace the possession and handling of the sample throughout its history, from collection to final disposition.

The samples will be delivered to the subcontractor under COC. As soon as the samples arrive at the subcontractor laboratory, the responsibility of those samples rests with the laboratory's designated "Sample Custodian" who is responsible for the receipt of all samples. The

subcontractor's Sample Custodian is responsible for transmitting the required documentation back to LANL (Sample Coordinator) acknowledging receipt of the samples and noting any problems/ resolutions. See Section II.A. for more details.

Each sample must always be under custody that is documented. A sample is under custody if

- It is in your possession; or
- It is in your view, after being in your possession; or
- It was in your possession and you locked it up; or
- It is in a designated secure area (accessible only to authorized persons)

The Chain-of-Custody records/documentation will become part of the SDG/RN File. A copy of the Chain-of-Custody documentation will be transmitted back to LANL by the data due date with the hard copy, raw data (and diskette, if required). See II.E. concerning Chain-of-Custody for sample return. The original Chain-of-Custody documentation will be transmitted with the samples when they are returned to LANL (See II.E.).

IX.B. Document Control Procedures - The subcontractor must have a document control program in place that assures that all documents for a specified SDG/RN of samples will be accounted for when the project is completed. Accountable documents include (but are not limited to) logbooks sample transmittal forms/COC records, sample worksheets, and any other documents relating to the sample and/or the analyses.

IX.C. SDG/RN File - The SDG/RN File (term to be used interchangeably with "RN file") has been mentioned in several sections of this document, and for purposes of this subcontract, means the original of the data package plus all records and documents related to the SDG/RN obtained or generated by the laboratory and not spelled out - either as hard copy or electronic - as a scheduled deliverable. In addition to the original data package, the SDG/RN File may consist of, but not be limited to, all chain-of-custody records, shipping documents (e.g., airbills), sample tags, benchsheets, logbook pages, screening records, instrument outputs, raw data, and telephone contact logs. This SDG/RN File must be transmitted to LANL with the hardcopy (and diskette, if required) by the data due date.

IX.D. Confidential Information - The subcontractor may (though unlikely) receive information from LANL identified as "confidential." This information must be handled separately only by authorized personnel, with all records stored in a separate locked file. Permission to reproduce any of this confidential information must be obtained from LANL.

X. KEY PERSONNEL FUNCTIONS - Each of the specific requirements for the routine analytical categories contains the specific key personnel functions that are unique to that analytical category. Herein are the additional key personnel functions that are required for any and all of these analytical categories. **Note: more than one function can be performed by a single person, except for the Quality Assurance Officer functions.** Equivalent experience may be substituted for specific educational requirements if it can be demonstrated.

1. Laboratory or Project manager - This person is responsible for the overall aspects of the subcontract and is the primary contact with LANL.

2. Sample Custodian - Responsible for receipt of LANL samples.

3. Document Control Officer - Responsible for all aspects of data deliverables - organization, packaging, copying, and delivery.

4. Quality Assurance Officer - This person must report independently to upper management and must not have any function directly involved in the generation of sample data for this RFP subcontract.

   Requires bachelor's degree in chemistry/science/ engineering or equivalent experience + 2 years experience including 1 year applied experience with QA principles and practices in an analytical laboratory.

5. Laboratory supervisors - all routine services - Requires a Bachelor;s degree in chemistry/science/engineering + 2 years of experience in the area supervised.

6. Instrument Operators for all routine services - Requires a Bachelor's degree in chemistry/science/engineering + one year of experience on the instrument (or all in equivalent experience).

7. Sample preparation specialists and other laboratory technicians - Requires a high school diploma + a course in general chemistry (college level) + one year of relevant experience (or all in relevant experience).

8. Systems Manager - Requires Bachelor's degree (or equivalent experience) with 4 courses in programming, information management, database management, or systems requirements analysis + 1 year experience in data or systems management, including 6 months with the programs in use in the laboratory.

9. Programmer Analyst - Requires Bachelor's degree (or equivalent experience) with 4 courses in programming, information management, database management, or systems requirements analysis + 1 year experience in systems or application programming, including 6 months with the programs in use by the laboratory.

10. Department of Transportation Hazardous Materials Regulations Expert - This role is in response to a DOE policy document stating that "...any contract placed with a contractor, subcontractor, agent, analytical laboratory, or similar organization expected to receive hazardous or radioactive materials must have on its staff an individual who is knowledgeable in Department of Transportation (DOT) hazardous materials regulations." *This applies also to shipment of samples (return to LANL) that may be hazardous or radioactive or found to be mixed wastes.* his individual(s) shall be identified in writing in any proposal submittal. A background statement of this individual's qualifications, along with copies of training certificates or any other documented source of training or establishment of knowledge of the DOT hazardous materials regulations, must accompany the proposal or bid." **Note:** this is a function, not a position, and can be carried out by any qualified staff member(s).

XI. SPECIAL COMMUNICATION - There may be instances when a laboratory's ability to meet a specific "EQL" or "EDL" on a sample is critical because of an action threshold (usually called a "screening action level - SAL" at LANL). The laboratory will be informed of this situation by Sample Management, with the request that the laboratory notify the Sample Coordinator when matrix problems impair the ability to meet that EQL/EDL.

XII. **Waste Management Policy** - The subcontractor laboratory must have a Waste Management policy in place meeting all relevant regulations.

SECTION III - SPECIFIC REQUIREMENTS FOR INORGANICS ANALYSES

I. Overview - Because of the history of weapons design at Los Alamos National Laboratory, dating back to the Manhattan Project in 1943, there is reason to believe that some sites/field units involved in DOE's Environmental Restoration Program may contain detectable concentrations of toxic inorganic trace elements that may have been used as tracers or spun off as byproducts during research and development activities for nuclear weapons.

The analytical data generated by the subcontractor under this subcontract will be used to determine if there are measurable concentrations of the targeted inorganics that will require remediation.

Sample matrices that the subcontractor may receive (but not limited to) could be water, waste water, soil, sludges, filters, and oils.

The methods cited in this section are methods of the United States Environmental Protection Agency (EPA). Target analytes are those required to meet New Mexico Environmental Division or U.S. EPA regulatory requirements. The methods generally consist of either TCLP extraction or an acid peroxide leach of the analytes from the sample matrix. In many cases, sample preparation methods are specific for a given analyte, matrix, and instrumental detection technique. Quantitation of the analytes involves the relationship of the response of the analyte in a sample to a response generated using standards.

II Target analytes/methods citations -

Following are the sample preparation procedures that are appropriate for use. The most recent version of SW-846 should be used. Alternatively, CLP sample preparation procedures (from Statement of Work ILM03.0) may be used, if appropriate for the matrix.

- SW-3005 Acid Digestion of waters for total recoverable or dissolved metals for analysis by flame AAS or ICP.
- SW-3010 Acid digestion of aqueous samples and extracts for total metals for analysis by flame AAS or ICP.
- SW-3020 Acid digestion of aqueous samples and extracts for total metals for analysis by furnace AAS, with the exception of As and Se, which are to be prepared according to methods 7060 and 7740.
- SW-3040 Dissolution procedure for oils, greases or waxes. ***Microwave digestion of these samples is preferred.***
- SW-3050 Acid digestion of sediments, sludges, and soils.
- SW-1311 Toxicity Characteristics Leaching Procedure (Note that changes made in the Federal Register, Volume 57, No 227, p. 55114, must be incorporated.
- SW-3015 and SW-3051 Microwave Digestion procedures.

Target Analyte Lists and detection limits:

Estimated Detection Limits (EDL) for water samples are based on CLP "Contract Required Detection Limits."

ICPAES (Recommended Method: SW - 6010A)

Analyte	Water EDL µg/L	Soil EDL mg/Kg
Aluminum	200	40
Antimony	60	12
Barium	200	40
Beryllium	5	1
Cadmium*	5	1
Calcium	5000	1000
Chromium*	10	2
Cobalt	50	10
Copper	25	5
Iron	100	20
Lead*	3	0.6
Magnesium	5000	1000
Manganese	15	3
Nickel	40	8
Potassium	5000	1000
Silver*	10	2
Sodium	5000	1000
Vanadium	50	10
Zinc	20	4

AA methods or ICP-MS may also be used for these analytes. In cases where the EDL cannot be met using ICPAES or false positives are known to be a problem historically within a laboratory, the subcontractor must use furnace AA techniques (e.g., method 7841 for thallium and method 7421 for lead) or ICP-MS (e.g. SW846 method 6020).

GFAA, ICP-MS

Analyte	Water EDL, µg/L	Soil EDL, mg/kg
Arsenic*	10	2
Lead*	1	0.2
Selenium*	5	1
Thallium	10	2

Cold Vapor AA

Analyte	Water EDL µg/L	Soil EDL mg/kg
Mercury *	0.2	0.1

SW-9010, 9010A, 9012, EPA 335.2

Analyte	EDL µg/L	EDL mg/kg
Total Cyanide	10	0.05

Soil EDLs for ICPAES, GFAA, and ICP-MS analytes are based upon a 1 gram sample taken to a final volume of 200 mL.

Soil EDLs for Cold Vapor AA are based upon a 0.2 gram sample taken to a final volume of 100 mL.

Soil EDLs for CLP ILM03.0 method 335.2 (CN) are based upon a 5 gram sample taken to a final volume of 250 mL.

The contractor may vary weights and final volumes for metals and cyanide analyses; however, any allowable variance must still meet the EDL

TCLP metals are identified with an asterisk (*) and may be requested as a separate determination. Laboratories should consider EDLs for TCLP metals using the Toxicity Characterization Leaching Procedure to be the regulatory limits. Method SW-1311 (7/92) is the method to be used for TCLP.

III. Reporting/Deliverables - There will be a combination of hardcopy and electronic deliverables, including raw data. Section II.B, Data delivery requirements, of the SOW describes the electronic deliverables for both initial and later terms.
The required hardcopy deliverables must be in the following order:

Raw data

- All raw data used to obtain the value for each reported result, including required QC measurements, instrument standardization, and sample analysis results. The order of the data should be ICPAES, ICP-MS, Furnace AA, Mercury, and Cyanide.

  Raw data must be labeled with LANL sample numbers.

- Digestion and distillation logs in the order of the raw data. These must include date, sample weights/volumes, information to identify which QC samples correspond to each batch digested, comments describing any significant sample changes or reactions which occur during preparation, and an indication of pH <2 or >12.

Soil sample results must be reported on a dry weight basis.

Note: All data must be legible and appropriately labelled.

IV. Quality Control (QC) requirements - It is important that the laboratory personnel follow Good Laboratory Practices throughout all operations involved in the analyses of samples for inorganics and that they maintain an internal QC program that is relevant to the analyses under consideration -in this case, inorganics.

Failure to meet acceptance criteria requires reanalysis of associated samples under acceptable criteria.

Acceptance criteria

The following quality control requirements must be performed and criteria met for all analyses performed under this subcontract.

1. Instrument Calibration

Guidelines for instrumental calibration are given in EPA 600/4-79-020. Instruments must be calibrated daily, or at minimum, each time the instrument is set up and used. The instrument standardization date and time must be included in the raw data.

For graphite furnace atomic absorption and ICP-MS systems, calibration standards should be prepared by diluting the stock metal solutions at the time of analysis. Date and time of preparation and analysis must be given in the raw data.

For atomic absorption systems, prepare a blank and at least three calibration standards in graduated amounts in the appropriate range. One atomic absorption calibration standard must be at the EDL. The calibration standards must be prepared using the same type of acid, or combination of acids, and at the same concentration as will result in the samples following sample preparation.

Beginning with the blank, aspirate or inject the standards and record the readings. Results for the calibration standards must be within 5% of the true value. Each standard's concentration and the calculations demonstrating that the 5% criterion has been met, must be provided with the raw data. If the values do not fall within this range, recalibration is necessary.

Calibration standards for AA, mercury, and cyanide procedures must be prepared so as to cover the expected calibration range at approximately equidistant intervals.

Baseline correction is acceptable as long as it is performed after every sample or after the continuing calibration verification (CCV - described in IV.2.b.) and blank check; resloping is acceptable as long as it is immediately preceded and immediately followed by a compliant CCV and continuing calibration blank (CCB - described in V.3.a). For ICP systems, calibrate the instrument according to instrument manufacturer's recommended procedures. At least two standards must be used for ICP calibration. One of the standards must be a blank.

2. Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)

a. Initial Calibration Verification (ICV)

Immediately after each of the instrument systems have been calibrated, the accuracy of the calibration shall be verified and documented for every analyte by the analysis of the contractor's certified ICV Solution(s). When measurements exceed the control limits of Table 1, the affected analytes must be reanalyzed after the problem has been corrected.

Where a certified solution of an analyte is not available from any source, analyses shall be conducted on an independent standard at a concentration other than that used for instrument calibration, but within the calibration range. An independent standard is defined as a standard composed of the analytes from a different source than those used in the standards for the instrument calibration.

For ICPAES, the ICV Solution(s) must be run at each wavelength used for analysis. For CN, the ICV standard must be distilled. The ICV for CN also serves as a Laboratory Control Sample; thus it must be distilled with the batch of samples analyzed in association with that ICV.

b. Continuing Calibration Verification (CCV)

To ensure calibration accuracy during each analytical run, one of the following standards are to be used for continuing calibration verification (CCV) and must be analyzed and reported for every wavelength used for the determination of each analyte, at a frequency of 10% or every 2 hours during an analytical run, whichever is more frequent. The ICV can be considered the first CCV in the run when calculating frequency. The standard must also be analyzed and reported after the last analytical sample. The analyte concentrations in the continuing calibration standard must be one of the following solutions at or near the mid-range levels of the calibration curve: (1) EPA Solutions, (2) NIST Standards, or (3) a Subcontractor-prepared standard solution.

Table 1

Initial and Continuing Calibration Verification Control Limits for Inorganic Analyses

Analytical Method	% of True Value Inorganic Species	Low Limit	High Limit
ICP/AA	Metals	90	110
Cold Vapor AA	Mercury	80	120
Other	Cyanide	85	115

The same continuing calibration standard must be used throughout the analytical run for a Sample Delivery Group.

Each CCV analyzed must reflect the conditions of analysis of all associated analytical samples (the preceding 10 analytical samples or the preceding analytical samples up to

the previous CCV). The duration of analyses, rinses and other related operations that may affect the CCV measured result may not be applied to the CCV to a greater extent than that applied to the associated analytical samples.

If the deviation of the continuing calibration verification is greater than the control limits specified in Table 1 - "Initial and Continuing Calibration Verification Control Limits for Inorganic Analyses", the preceding 10 analytical samples, or all analytical samples analyzed since the last compliant calibration verification must be reanalyzed for the analytes affected.

3. Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Preparation Blank (PB) Analyses

a. Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) Analyses

A calibration blank must be analyzed at each wavelength used for analysis immediately after every initial and continuing calibration verification at a frequency of 10% or every 2 hours during the run, whichever is more frequent. The blank must be analyzed at the beginning of the run and after the last analytical sample. If the absolute value of the blank result exceeds the EDL, reanalyze the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration blank.

b. Preparation Blank (PB) Analysis

At least one preparation blank (or reagent blank), consisting of deionized distilled water processed through each sample preparation and analysis procedure must be prepared and analyzed with every Sample Delivery Group (SDG/RN), or with each batch of samples digested, whichever is more frequent.

Since a suitable blank matrix may not be available for soils or solid materials, the contractor may use the same water, weighed out as though it were a soil or solid, as that used for the preparation of the standards and samples for the PB.

If suitable blank matrix material for soil becomes available it should be used instead of water.

The first batch of samples in an SDG/RN is to be assigned to preparation blank number 1, the second batch of samples to preparation blank number 2, etc. Each data package must contain the results of all the preparation blank analyses associated with the samples in that SDG/RN.

This blank is to be reported for each SDG/RN and used in all analyses to ascertain whether sample concentrations reflect contamination in the following manner:

1) If the absolute value of the concentration of the blank is less than or equal to the EDL, no correction of sample results is performed.

2) If any analyte concentration in the blank is above the EDL, the lowest concentration of that analyte in the associated samples must be 10x the blank concentration. Otherwise, all samples associated with the blank with the analyte's concentration less than 10x the blank concentration and above the EDL, must be redigested and reanalyzed for that analyte (except for an identified field blank). The sample concentration is not to be corrected for the blank value.

3) If the concentration of the blank is below the negative EDL, then all samples reported below 10x EDL associated with the blank must be redigested and reanalyzed.

4. ICP Interference Check Sample (ICS) Analysis

To verify interelement and background correction factors, the Subcontractor must analyze and report the results for the ICP Interference Check Sample at the beginning of each analytical run, but not before Initial Calibration Verification.

Results for the ICP analysis of the ICS during the analytical runs must fall within the control limit of ±20% of the true value for the analytes included in the Interference Check sample. If not, reanalyze the analytical samples analyzed since the last acceptable ICS.

If true values for analytes contained in the ICS and analyzed by ICP are not supplied with the ICS, the mean must be determined by initially analyzing the ICS at least five times repetitively for the particular analytes. This mean determination must be made during an analytical run where the results for the previously supplied ICS met all subcontract specifications. Additionally, the result of this initial mean determination is to be used as the true value for the lifetime of that solution (i.e., until the solution is exhausted).

Independent ICP Check Samples must be prepared with interferent and analyte concentrations at the levels specified in Table 2-Interferent and Analyte Elemental Concentrations Used for ICP Interference Check Sample. This is the minimum set of interferences that must be compensated for. Due to variation in sample matrices, other interferences may be present. Subcontractor laboratories are encouraged to correct for as many interferences as practicable with their ICP instrument.

The mean value and standard deviation must be established by initially analyzing the Check Samples at least five times repetitively for each parameter. Results must fall within the control limit of ±20% of the established mean value. The mean and standard deviation must be reported in the raw data.

TABLE 2. Interferent and Analyte Elemental Concentrations used for ICP Interference Check Sample

Analytes	(mg/L)	Interferents	(mg/L)
Ag	1.0	Al	500
Ba	0.5	Ca	500
Be	0.5	Fe	200
Cd	1.0	Mg	500
Co	0.5		
Cr	0.5		
Cu	0.5		
Mn	0.5		
Ni	1.0		
Pb	1.0		
V	0.5		
Zn	1.0		

5. Spike Sample Analysis (S)

The spike sample analysis is designed to provide information about the effect of the sample matrix on the digestion and measurement methodology. The spike is added before the digestion (i.e., prior to the addition of other reagents) and prior to any distillation steps (i.e., CN). At least one spike sample analysis must be performed on each group of samples of a similar matrix type (i.e., water, soil) for each Sample Delivery Group.

If the spike analysis is performed on the same sample that is chosen for the duplicate sample analysis, spike calculations must be performed using the results of the sample designated as the "original sample" (see section 6, Duplicate Sample Analysis). The average of the duplicate results cannot be used for the purpose of determining percent recovery. Samples identified as field blanks cannot be used for spiked sample analysis. LANL may require that a specific sample be used for the spike sample analysis.

The analyte spike must be added in the amount given in Table 3-Spiking Levels for Spike Sample Analysis, for each element analyzed. Note: See Table 3 footnotes for concentration levels and applications. If two analytical methods are used to obtain the reported values for the same element within a Sample Delivery Group (i.e., ICP, GFAA), spike samples must be run by each method used.

The spike recovery should be within the advisory limits of 75-125%. An exception to this rule is granted in situations where the sample concentration exceeds the spike concentration by a factor of four or more.

Individual component percent recoveries (%R) are calculated as follows:

$$\% \text{ Recovery} = \frac{(SSR - SR)}{SA} \times 100$$

where SSR = Spiked Sample Result,
SR = Sample Result, and
SA = Spike Added.

When sample concentration is less than the instrument detection limit, use SR = 0 only for purposes of calculating percent recovery.

The units for reporting spike sample results will be identical to those used for reporting sample results (i.e., µg/L for aqueous and mg/Kg dry weight basis for solid).

TABLE 3. SPIKING LEVELS FOR SPIKE SAMPLE ANALYSIS

	For ICP/AA		For Furnace AA		
Element	Water, µg/L	Soil, (2) mg/kg	Water, µg/L	Soil,(2) mg/kg	Other (1)(2)
Aluminum	2,000	*			
Antimony	500	100	100	20	
Arsenic	2,000	400	40	8	
Barium	2,000	400			
Beryllium	50	10			
Cadmium	50	10	5	1	
Calcium	*	*			
Chromium	200	40			
Cobalt	500	100			
Copper	250	50			
Iron	1,000	*			
Lead	500	100	20	4	
Magnesium	*	*			
Manganese	500	100			
Mercury					1
Nickel	500	100			
Potassium	*	*			
Selenium	2,000	400	10	2	
Silver	50	10			
Sodium	*	*			
Thallium	2,000	400	50	10	
Vanadium	500	100			
Zinc	500	100			
Cyanide					100 (3)

*No spike required. **NOTE:** Elements without spike levels and not designated with an asterisk, must be spiked at appropriate levels.

[1]Spiking levels reported are for both water and soil/sediment matrices.

[2]The levels shown indicate concentrations in the final digestate of the spiked sample (100 mL for mercury and 200 mL for all other metals) when the wet weight of 1 gram (for ICP, Furnace, and Flame AA), or 0.2 grams (for mercury) of sample is taken for analysis. Adjustment must be made to maintain these spiking levels when the weight of sample taken deviates by more than 10% of these values. Appropriate adjustment must be made for microwave digestion procedure where 0.5 grams of sample or 50.0 mL (45.0 mL of sample plus 5.0 mL of acid) of aqueous sample are required for analysis.

[3]The level shown indicates the amount of cyanide that must be added to the original (undistilled) sample. For instance, 100 µg must be added per each liter of aqueous sample.

For soil samples, 25 µg of cyanide must be added per each gram of solid sample taken for analysis. If the final distillate volume is 250 mL, then the distillate will contain cyanide at a concentration of 100 µg/L.
Assuming a sample of one gram, the manual and semi-automatic colorimetric methods call for a cyanide concentration of 25 µg per the 500 mL mixture of the sample, reagents, and water before distillation. The final distillate, in this case, contains cyanide at a concentration of 100 µg/L. For the midi-distillation method, a cyanide concentration of 25 µg must be added into the 50 mL mixture of sample, reagents, and water before distillation. This yields a cyanide concentration of 500 µg/L in final distillate of 50 mL.

6. Duplicate Sample Analysis (D)

One duplicate sample must be analyzed from each group of samples of a similar matrix type (i.e., water, soil) for each Sample Delivery Group. Duplicates cannot be averaged for reporting.

The relative percent differences (RPD) for each component are calculated as follows:

$$RPD = \frac{(S - D)}{(S + D)} \times 200$$

where RPD = relative percent difference,
S = first sample value (original), and
D = second sample value (duplicate).

The results of the duplicate sample analyses must be reported in µg/L for aqueous samples and mg/kg dry weight basis for solid original and duplicate samples. An advisory control limit of 20% for RPD is appropriate for original and duplicate sample values

greater than or equal to 5x EDL. A control limit of (±) the EDL must be used if either the sample or duplicate value is less than 5x the EDL.

If one result is above the 5x EDL level and the other is below, use the ± EDL criteria. If both sample values are less than the MDL (see # 8), the RPD is not calculated. For solid sample or duplicate results > 5x EDL, the absolute value of the EDL is corrected for sample weight and percent solids. The percent difference data will be used by LANL to evaluate the long-term precision of the methods for each parameter.

7. Laboratory Control Sample (LCS) Analysis

An aqueous Laboratory Control Sample (LCS) must be analyzed for each analyte using the same sample preparations, analytical methods and QA/QC procedures employed for the LANL samples received. One aqueous LCS must be prepared and analyzed for every group of aqueous samples in a Sample Delivery Group, or for each batch of aqueous samples digested, whichever is more frequent.

If a certified LCS soil or solid matrix is available from a vendor, it may be used. Otherwise, the contractor must use the aqueous LCS for soil or solid matrices. Should the later case prevail, the contractor must treat the aqueous LCS solution as a soil or solid material, i.e., it must be weighed out, prepared and reported as if it were a soil or solid matrix.

If the percent recovery for the aqueous LCS falls outside the control limits of 80-120% (exception: Ag and Sb), the analyses must be terminated, the problem corrected, and the samples associated with that LCS redigested and reanalyzed.

8. Method Detection Limit (MDL) Determination

Before any field samples are analyzed under this subcontract, the method detection limits (in µg/L) must be determined for each instrument used, within 30 days of the start of subcontract analyses and at least annually thereafter, and must be less than the EDL.

The Method Detection Limits (in µg/L) shall be determined by multiplying by 3, the average of the standard deviations obtained on three nonconsecutive days from the analysis of a preparation blank, with seven consecutive measurements per day. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate sample). MDL's must be determined and reported for each wavelength used in the analysis of the samples.

The annually-determined MDL for an instrument must always be used as the MDL for that instrument during that year. If the instrument is adjusted in any way that may affect the MDL, the MDL for that instrument must be redetermined and the results submitted for use as the established MDL for that instrument for the remainder of the year.

If multiple instruments are used for the analysis of an element within a Sample Delivery Group, the highest MDL must be used for reporting concentration values for that Sample Delivery Group.

9. Interelement Corrections for ICP

Before any field samples are analyzed under this subcontract, the ICP interelement correction factors must be determined prior to the start of subcontract analyses and at least annually thereafter. Correction factors for spectral interference due to Al, Ca, Fe, and Mg must be determined for all ICP instruments at all wavelengths used for each analyte reported by ICP. Correction factors for spectral interference due to analytes other than Al, Ca, Fe, and Mg must be reported if they were applied.

If the instrument was adjusted in any way that may affect the ICP interelement correction factors, the factors must be redetermined and the results submitted for use.

10. Analytical Range Verification (ARV)

The analyte concentrations in this standard represent the upper limit of the ICP analytical range beyond which results cannot be reported under this subcontract without dilution of the analytical sample. These concentrations may be higher than the highest calibration standard. If no concentrations higher than the highest calibration standard (actual instrument reading, before any dilution correction) will be reported, this standard need not be run. If, however, the useful range of the instrument extends beyond the calibration range, an analytical range verification check standard should be analyzed and reported for each element. The standard must be analyzed during a routine analytical run performed under this subcontract, and the analytically determined concentration of this standard must be within 5% of the true value.

11. Furnace Atomic Absorption (AA) QC Analyses

All furnace analyses must fall within the calibration range. In addition, all analyses will require duplicate injections. The absorbance or concentration of each injection must be reported in the raw data as well as the average absorbance or concentration values and the relative standard deviation (RSD). Average concentration values are used for reporting purposes. A maximum of 10 full sample analyses to a maximum 20 injections may be performed between each consecutive calibration verification and blank. For concentrations greater than the EDL, the duplicate injection readings must agree within 20% RSD, or the analytical sample must be rerun once (i.e., two additional burns).

SECTION III - SPECIFIC REQUIREMENTS FOR VOLATILE ORGANIC ANALYSES

I. Overview - Because of the history of weapons design at Los Alamos National Laboratory, dating back to the Manhattan Project in 1943, there is reason to believe that some sites/field units involved in DOE's Environmental Restoration Program may contain detectable concentrations of volatile organic solvents that may have been used during research and development of nuclear weapons, conventional weapons and novel materials.

The analytical data generated by the subcontractor under this subcontract will be used to determine if there are measurable concentrations of the targeted volatile organic compounds that will require remediation or long term monitoring.

Sample matrices that the subcontractor may receive include, but are not limited to water, waste water, soil, sludges, traps, filters, and oils.

The methods cited in this section are methods of the United States Environmental Protection Agency (U.S EPA). Target compounds are those required to meet New Mexico Environment Division or U.S. EPA regulatory requirements.

II. Target Compounds/Methods Citations

Estimated Quantitation Limits (EQLs)-

Volatile Organic Compounds	Water µg/L	Soil/Solids µg/kg
Chloromethane	10	10
Vinyl Chloride	10	10
Bromomethane	10	10
Chloroethane	10	10
Acetone	20	20
Dichlorodifluoromethane	10	10
Iodomethane	5	5
Trichlorotrifluoroethane	5	5
Trichlorofluoromethane	5	5
Methylene Chloride	5	5
1,1-Dichloroethene	5	5
Carbon Disulfide	5	5
1,1-Dichloroethane	5	5
1,2-Dichloroethene (total)	10	10
Bromochloromethane	5	5
Chloroform	5	5

1,2-Dichloroethane	5	5
1,1-Dichloropropene	5	5
2-Butanone	20	20
2,2-Dichloropropane	5	5
1,1,1-Trichloroethane	5	5
Carbon Tetrachloride	5	5
Benzene	5	5
1,2-Dichloropropane	5	5
Trichloroethene	5	5
Dibromomethane	5	5
Bromodichloromethane	5	5
t-1,3-Dichloropropene	5	5
c-1,3-Dichloropropene	5	5
1,1,2-Trichloroethane	5	5
1,3-Dichloropropane	5	5
Chlorodibromomethane	5	5
4-Methyl-2-Pentanone	20	20
Toluene	5	5
2-Hexanone	20	20
1,2-Dibromoethane	5	5
Tetrachloroethene	5	5
Chlorobenzene	5	5
1,1,1,2-Tetrachloroethane	5	5
Ethylbenzene	5	5
o,m,p-Xylene (mixed)	5	5
Styrene	5	5
Bromoform	5	5
1,1,2,2,-Tetrachloroethane	5	5
1,2,3-Trichloropropane	5	5
Isopropylbenzene	5	5
Bromobenzene	5	5
n-Propylbenzene	5	5

2-Chlorotoluene	5	5
4-Chlorotoluene	5	5
1,3,5-Trimethylbenzene	5	5
tert-Butylbenzene	5	5
1,2,4-Trimethylbenzene	5	5
sec-Butylbenzene	5	5
1,3-Dichlorobenzene	5	5
1,4-Dichlorobenzene	5	5
p-Isopropyltoluene	5	5
1,2-Dichlorobenzene	5	5
n-Butylbenzene	5	5
1,2-Dibromo-3-Chloropropane	10	10

Tentatively Identified Compounds (TICs) may be requested. If requested, they should be identified and quantitated per the CLP method for volatiles, OLM02.0 (or more recent).

Methods

The U.S. EPA methods that are options for use are method SW-8260 (11/90 or more recent) or the CLP method for volatiles (OLM02.0 or more recent, using capillary column). These methods are based on purge and trap sample extraction/concentration followed by gas chromatography/mass spectrometry analysis.

Note: Quality control requirements are specified in Section IV, regardless of the method selected.

For medium level analysis - At times, a medium level or methanol extraction method will occur in the field. When it does occur, the laboratory will receive the methanol sample extract as the matrix to be analyzed. The extract is to be analyzed within 40 days from the time the extraction occurred. A benchsheet containing the sample amount extract, solvent volume added, and the extraction date will accompany the sample extract(s). In addition to the sample extract, the laboratory will receive a sampling vial containing a portion of the methanol used for the extraction. An aliquot of the methanol will be injected into reagent water and analyzed as the method blank. The analysis of the sample extract(s) and method blank will happen under the aqueous calibration standards and analytical conditions. As a result of the medium level method, the EQL's will be elevated.

III. **Reporting/Deliverables** - There will be a combination of hardcopy and diskette deliverables. Section II.B, Data delivery requirements, of the Statement of Work describes the electronic deliverables for both initial and later term.

The hardcopy deliverables are as follow and must be in the following order, chronologically arranged, by instrument:

Sample Data

- Chromatogram and quantitation report for each sample

- Raw spectra, background-subtracted spectra, and reference spectra for target compounds that are identified in the sample. The subcontractor may be asked to provide these same spectra for compounds that have been noted as false positives, upon occasion.

- If TICs are requested, copies of mass spectra of TICs identified with the associated best-match spectra (3 spectra).

- Sample data (chromatograms, quantitation reports, and spectra) for all re-analyses.

Standards Data

- Chromatogram and quantitation report for all standards associated with the initial and continuing calibrations.

- For a period of time until the LANL EDD includes complete electronic reporting capability , the following organics hardcopy deliverables are required in addition (See last paragraph of II.B of the "General Requirements"):

- CLP volatiles forms 6, 7, and 8 (initial and continuing calibration standards and internal standard QC summary)

Raw QC data

For bromofluorobenzene (BFB), for each 12-hour period, for each GC/MS instrument:

- Mass listing - % relative abundance

- For a period of time until the LANL EDD includes complete electronic reporting capability , the following organics hardcopy deliverables are required in addition (See last paragraph of II.B of the "General Requirements"):

- CLP form 5 computer-generated report.

Blank data - in chronological order.

- Chromatograms and quantitation reports

- Raw spectra, background-subtracted spectra, and reference standard spectra for target compounds identified and any compounds noted as false positives by the analyst.

Matrix spikes/duplicate matrix spikes - if they are requested, chromatograms and quantitation reports are required.

Laboratory Control Samples (LCS) - for LCSs analyzed in association with LANL samples, the chromatograms and quantitation reports are required.

Data must be reported on a dry weight basis.

pH for all aqueous samples for volatiles analysis must be recorded and provided with hardcopy until such time as it is incorporated into the spreadsheet.

Note: All hardcopy sample data must be legible and clearly labeled with the LANL sample number, lab file ID, date and time of analysis, and GC/MS instrument ID. Compound names must be clearly marked on all spectra. Hardcopy standards data and raw QC data must be legible and clearly labeled with the lab file ID, date and time of analysis, and GC/MS instrument ID.

IV. Quality Control (QC) requirements - It is important that the laboratory personnel follow Good Laboratory Practices throughout all operations involved in the analyses of samples. Refer to SW-846 chapter one for general quality control requirements for volatile organic analysis. **Failure to meet criteria requires reanalysis of associated samples under acceptable criteria.**

Matrix spike and matrix spike duplicate analysis will be requested if needed. A sample will be submitted to be used as the matrix media if matrix spikes are requested, with direction on analytes to spike.

Acceptance Criteria

1. Instruments Performance Check -

The mass calibration and resolution are verified by the analysis of the instrument performance check solution, p-Bromofluorobenzene (BFB). The instrument performance check solution, BFB, must be injected at the beginning of each 12 hour period of analysis and must meet the following ion abundance criteria:

Mass	Intensity Required (Relative Abundance)
50	8.0 to 40.0% of mass 95
75	30.0 to 66.0% of mass 95
95	base peak, 100% relative abundance
96	5.0 to 9.0% of mass 95
173	less than 2.0% of mass 174
174	50.0 to 120.0% of mass 95
175	4.0 to 9.0% of mass 174
176	93.0 to 101.0% of mass 174
177	5.0 to 9.0% of mass 176

2. Initial and Continuing Calibration -

Prepare 5 aqueous initial calibration standard solutions containing all of the purgeable target compounds and /system monitoring compounds (also referred to as "surrogates") at 10, 20, 50, 100, and 200 µg/mL concentration. It is required that all 3 of the xylene

isomers (o ,m, p) be in the calibration standards at concentrations of each isomer equal to that of the other target compounds. Similarly, the cis and trans isomers of 1,2-dichloroethene must both be present in the standards at concentrations equal to the other target analytes.

Separate calibrations (initial and continuing) must be performed for aqueous and solid matrices with the aqueous calibration standards undergoing an unheated purge and the solid calibration standards undergoing a heated purge. Sample extracts of a medium level method can be analyzed using the calibration for aqueous matrices undergoing an unheated purge.

If time remains in the 12-hour time period after meeting the acceptance criteria for the initial calibration, samples may be analyzed. It is not necessary to analyze a continuing calibration standard if the initial calibration standard that is the same concentration as the continuing calibration standard meets the continuing calibration acceptance criteria. (i.e. a CLP form 7A must be generated). If time does not remain, a new injection of BFB must meet the abundance criteria given in item 1 followed by a continuing calibration standard.

The relative response factor (RRF) at each concentration level for each target and system monitoring compound must be greater or equal to the compound's minimum acceptable response factor listed in table 1.

The % RSD for each target and system monitoring compound from the initial calibration must be less than or equal to the Maximum % RSD listed in Table 1.

The relative response factor percent difference (%D) for each target and system monitoring compound must be less than or equal to the value listed in Table 1.

Up to two compounds may fail to meet the acceptance criteria. However, these compounds must have a minimum RRF greater than or equal to 0.010, and the % RSD or % Diff must be less than or equal to 40.0%.

If a target compound is detected in a sample for which the continuing calibration % Diff >25% for that compound, the sample must be reanalyzed under a continuing calibration with an acceptable % Diff for that compound.

3. Internal Standards -

The minimum required internal standards are chlorobenzene-d5, 1,4-diflourobenzene, and 1,4-dichlorobenzene-d4. A retention time and response check must be performed on every internal standard for samples analyzed. The retention time (RT) for an internal standard from a sample cannot > ± 30 seconds from the previous daily calibration The response area of an internal standard from a sample cannot exceed a factor of 2 (-50% to +100%) from the previous daily calibration.

4. Surrogates or System Monitoring Compounds -

The minimum required surrogate compounds are toluene-d8, bromofluorobenzene, and dibromofluoromethane. Required recovery limits follow

	Water	Soil
Toluene-d8	88-110	81-117
Bromofluorobenzene	86-115	74-121
Dibromofluoromethane	86-118	80-120

5. Method Blanks -

A separate method blank must be extracted and/or analyzed for each method, matrix and /or analytical run prior to sample analysis. The method blank must be analyzed under the same conditions as required for the associated matrix. For example, the method blank for a solid sample matrix must undergo a heated purge. A method blank associated with a medium level solid analysis is to be injected with 100 ul of the methanol used for the medium level extraction.

Volatile organic target compounds must not be present in the blank at a concentration > the estimated quantitation limits (EQLs) with the exception of acetone, methylene chloride, and 2-butanone. Acetone, methylene chloride, and 2-butanone can be present at up to five times the EQL before corrective action is required. Sample results must not be corrected by subtracting any method blank value.

An instrument blank must be analyzed after any sample that has had a target compound two times the linear range of the instrument (400 ng/ml) to check for carry-over.

6. Samples

Sample (s) must be reanalyzed when one or more of the following occur:

- Acceptance criteria are not met
- Carry-over or laboratory contamination is suspected
- A target compound is over the instruments linear range (200 ng/ml). In this case. a dilution is required.

It is important that the lowest possible EQL's are achieved. If the initial analysis of a sample was performed at a dilution and there were no target compounds and/or TICs detected above the EQL, a re-analysis at a higher concentration must occur unless the reanalysis cannot be performed at a level five times the initial diluted analysis. For example, if the initial analysis was diluted 20% with no target and/TICs detected, a reanalysis without a dilution can be performed. However, if the sample was diluted 25 or 50%, a reanalysis is not necessary and the diluted initial analysis is reported.

When a reanalysis occurs, both sets of data are to be included.

TABLE 1
Acceptance Criteria for Initial and Continuing Calibration of Target and System Monitoring Compounds

Target Compound	Minimum RRF	Maximum % RSD	Maximum % Diff
Chloromethane	0.010	None	None
Bromomethane	0.100	20.5	±25.0
Vinyl chloride	0.100	20.5	±25.0
Chloroethane	0.010	None	None
Methylene chloride	0.010	None	None
Acetone	0.010	None	None
Dichlorodifluoromethane	0.010	None	None
Iodomethane	0.010	None	None
Trichlorotrifluoroethane	0.010	None	None
Trichlorofluoromethane	0.010	None	None
Carbon disulfide	0.010	None	None
1,1-Dichloroethane	0.100	20.5	±25.0
1,1-Dichloroethene	0.200	20.5	±25.0
1,2-Dichloroethene (total)	0.010	None	None
Bromochloromethane	0.010	None	None
Chloroform	0.200	20.5	±25.0
1,2-Dichloroethane	0.100	20.5	±25.0
1,1-Dichloropropene	0.010	None	None
2-Butanone	0.010	None	None
2,2-Dichloropropane	0.010	None	None
1,1,1-Trichloroethane	0.100	20.5	±25.0
Carbon tetrachloride	0.100	20.5	±25.0
Bromodichloromethane	0.200	20.5	±25.0
1,2-Dichloropropane	0.010	None	None
cis-1,3-Dichloropropene	0.200	20.5	±25.0
Trichloroethene	0.300	20.5	±25.0

Table 1

Acceptance Criteria for Initial and Continuing Calibration of Target and System Monitoring Compounds (cont.)

Target Compound	Minimum RRF	Maximum %RSD	Maximum %Diff
Dibromomethane	0.010	None	None
Dibromochloromethane	0.100	20.5	±25.0
1,1,2-Trichloroethane	0.100	20.5	±25.0
Benzene	0.500	20.5	±25.0
trans-1,3-Dichloropropene	0.100	20.5	±25.0
1,3-Dichloropropane	0.100	20.5	±25.0
4-Methyl-2-pentanone	0.010	None	None
Bromoform	0.100	20.5	±25.0
2-Hexanone	0.010	None	None
1,2-Dibromoethane	0.100	20.5	±25.0
Tetrachloroethene	0.200	20.5	±25.0
1,1,2,2,-Tetrachloroethane	0.300	20.5	±25.0
Toluene	0.400	20.5	±25.0
Chlorobenzene	0.500	20.5	±25.0
1,1,1,2-Tetrachloroethane	0.100	20.5	±25.0
Ethylbenzene	0.100	20.5	±25.0
Styrene	0.300	20.5	±25.0
Xylenes (Mixed)	0.300	20.5	±25.0
1,2,3-Trichloropropene	0.100	20.5	±25.0
Isopropylbenzene	0.100	20.5	±25.0
Bromobenzene	0.100	20.5	±25.0
n-Propylbenzene	0.100	20.5	±25.0
2-Chlorotoluene	0.100	20.5	±25.0
4-Chlorotoluene	0.100	20.5	±25.0
1,3,5-Trimethylbenzene	0.100	20.5	±25.0

tert-Butylbenzene	0.100	20.5	±25.0

Table 1

Acceptance Criteria for Initial and Continuing Calibration of Target and System Monitoring Compounds (cont.)

Target Compound	Minimum RRF	Maximum %RSD	Maximum % Diff
1,2,4-Trimethylbenzene	0.100	20.5	±25.0
sec-butylbenzene	0.100	20.5	±25.0
1,3-Dichlorobenzene	0.100	20.5	±25.0
1,4-Dicholorbenzene	0.100	20.5	±25.0
p-isopropyltoluene	0.100	20.5	±25.0
1,2-Dichlorobenzene	0.100	20.5	±25.0
n-Butylbenzene	0.100	20.5	±25.0
1,2-Dibromo-3-chloropropane	0.010	None	None
System Monitoring Compounds			
Bromofluorobenzene	0.200	20.5	±25.0
Toluene-d8	0.010	None	None
Dibromofluoromethane	0.010	None	None

SECTION III - SPECIFIC REQUIREMENTS FOR SEMIVOLATILE ANALYSES

I. Overview - Because of the history of weapons design at Los Alamos National Laboratory, dating back to the Manhattan Project in 1943, there is reason to believe that some sites/field units involved in DOE's Environmental Restoration Program may contain detectable concentrations of semi-volatile organics that may have been used during research and development of nuclear weapons, conventional weapons and novel materials.

The analytical data generated by the subcontractor under this subcontract will be used to determine if there are measurable concentrations of the targeted semi-volatile organic compounds that will require remediation or long term monitoring.

Sample matrices that the subcontractor may receive include, but are not limited to water, waste water, soil, sludges, traps, filters, and oils.

The methods cited in this section are methods of the United States Environmental Protection Agency (U.S EPA). Target compounds are those required to meet New Mexico Environment Division or U.S. EPA regulatory requirements.

II. Target Compounds/Method Citations

Estimated Quantitation Limits (EQLs) for Semivolatile Organics

SEMIVOLATILE ORGANIC COMPOUND	Water µg/L	Soil/Solid µg/kg*
Acenaphthene	10	330
Acenaphthylene	10	330
Aniline	20	660
Anthracene	10	330
Azobenzene	20	660
Benzo(a)anthracene	10	330
Benzoic acid	50	3300
Benzo(b)flouranthene	10	330
Benzo(K)flouranthene	10	330
Benzo(g,h,i)perylene	10	330
Benzo(a)pyrene	10	330
Benzyl alcohol	20	1300
Bis(2-chloroethoxy)methane	10	330
Bis(2-chloroethyl)ether	10	330
4-Bromophenyl phenylether	10	330

Butylbenzylphthalate	10	330
4-Chloroaniline	20	1300
4-Chloro-3-methylphenol	20	630
2-Chloronaphthalene	10	330
2-Chlorophenol	10	330
4-Chlorophenyl phenylether	10	330
Chrysene	10	330
Dibenz(a,h)anthracene	10	330
Dibenzofuran	10	330
1,2-Dichlorobenzene	10	330
1,3-Dichlorobenzene	10	330
1,4-Dichlorobenzene	10	330
3,3'-Dichlorobenzidine	20	660
2,4-Dichlorophenol	10	330
Diethylphthalate	10	330
Dimethyl phthalate	10	330
2,4-Dimethylphenol	10	330
2,4-Dinitrophenol	50	1600
Di-n-butylphthalate	10	330
4,6-Dinitro-2-methylphenol	50	1600
2,4-Dinitrotoluene	10	330
2,6-Dinitrotoluene	10	330
Di-n-octyl phthalate	10	330
Bis(2-ethylhexyl)phthalate	10	330
Fluoranthene	10	330
Fluorene	10	330
Hexachlorobenzene	10	330
Hexachlorobutadiene	10	330
Hexachlorocyclopentadiene	10	330
Hexachloroethane	10	330
Indeno(1,2,3-cd)pyrene	10	330
Isophorone	10	330

2-Methylnaphthalene	10	330
2-Methylphenol	10	330
4-Methylphenol	10	330
Naphthalene	10	330
2-Nitroaniline	50	1600
3-Nitroaniline	50	1600
4-Nitroaniline	20	660
Nitrobenzene	10	330
2-Nitrophenol	10	330
4-Nitrophenol	50	1600
N-Nitrosodimethylamine	10	330
N-Nitrosodiphenylamine	10	330
N-Nitroso-di-n-propylamine	10	330
2,2'-oxybis(1-Chloropropane)	10	330
Pentachlorophenol	50	1600
Phenanthrene	10	330
Phenol	10	330
Pyrene	10	330
1,2,4-Trichlorobenzene	10	330
2,4,5-Trichlorophenol	50	1600
2,4,6-Trichlorophenol	10	330

- EQLs for soil are based on no GPC being performed. The laboratories' GPC equipment will determine what the EQL is, based on the volume of extract the GPC equipment uses. However, if it is possible, in order to provide the lowest possible quantitation limits, the laboratories should concentrate the GPC extract to a volume that makes the EQL for a sample that underwent GPC clean-up no more than twice the listed EQL.

  ND = not determined, according to SW-8270

Tentatively Identified Compounds (TICs) may be requested. If requested, they should be identified and quantitated per the CLP method for semivolatiles, OLM02.0 (or more recent).

Methods

The U.S. EPA methods that are options for use are method SW-8270 (11/90 or more recent) or the CLP method for semivolatiles (OLM02.0 or more recent). These methods are based on solvent extraction, concentration and GC/MS detection and quantitation.

Note: Quality control requirements are specified in Section IV, regardless of the method selected.

Methods 3510, 3520, 3540 or 3550 can be used for sample preparation for semi-volatile organic compounds. An overview of these extraction methods and guidance for method selection can be found in method 3500. Gel permeation chromatography (Method 3640 or CLP) and/or other cleanup procedures must be performed when the nature of the sample requires it (SW-3600 provides guidance on cleanup usage/procedures).

III. Reporting/Deliverables - There will be a combination of hardcopy and diskette deliverables. Section II.B, Data delivery requirements, of the SOW, describes the electronic deliverables for both initial and later term.

The hardcopy deliverables are as follow and must be in the following order, chronologically arranged, by instrument:

Sample Data

- Chromatogram and quantitation report for each sample
- Raw spectra, background-subtracted spectra, and reference standard spectra for target compounds. The subcontractor may be asked to provide these spectra for compounds noted as false positives by the analyst.
- If TICs are requested, copies of mass spectra of TICs identified with the associated best-match spectra (3 spectra).
- Sample data (chromatograms, quantitation reports, and spectra) for all re-analyses.

Standards Data

- Chromatograms and quantitation reports for all standards associated with the initial and continuing calibration.
- For a period of time until the LANL EDD includes complete electronic reporting capability, the following organics hardcopy deliverables are required in addition (See last paragraph of II.B of the "General Requirements"):
  - CLP forms 6,7,and 8 for semivolatiles (initial and continuing calibration standards and internal standard qC summary)

Raw QC data

For decafluorotriphenylphosphine (DFTPP), for each 12-hour period, for each GC/MS instrument:

- Mass listing - % relative abundance

Blank data - in chronological order.

- Chromatograms and quantitation reports

- For a period of time until the LANL EDD includes complete electronic reporting capability, the following organics hardcopy deliverables are required in addition (See last paragraph of II.B of the "General Requirements"):

  - CLP form 5 computer generated report

- Raw spectra, background-subtracted spectra, and reference standard spectra for identified target compounds, including those for compounds noted as false positives by the analyst.

- If TICs are requested and TICs are found in the blank, include the GC/MS library search spectra.

Matrix spikes/matrix spike duplicates - If these are requested the subcontractor will be provided with instructions for spiking.

Laboratory Control Samples (LCS) - for LCSs analyzed in association with LANL samples, the chromatograms and quantitation reports are required.

Extraction benchsheets including GPC standard data if GPC was performed.

Results, including EQLs, must be reported on a dry weight basis.

Note: All hardcopy sample data must be legible and clearly labeled with the LANL sample number, lab file ID, date and time of analysis, and GC/MS Instrument ID. Compound names must be clearly marked on all spectra. Hardcopy standards data and raw QC data must be legible and clearly labeled with the lab file ID, date and time of analysis, and GC/MS Instrument ID.

IV. Quality Control (QC) requirements - It is important that the laboratory personnel follow Good Laboratory Practices throughout all operations involved in the analyses of samples. Refer to SW-846 chapter one for general quality control requirements for semivolatile organic analysis. In addition, the QC requirements listed under "Acceptance Criteria" must be followed and criteria met. **Failure to meet criteria requires reanalysis of associated samples under acceptable criteria.**

Matrix spike and matrix spike duplicate analysis will be requested if needed. A sample will be submitted to be used as the matrix media if matrix spikes are requested, in instructions for compounds to spike will be provided.

Acceptance Criteria

1. Instruments Performance Check -

The mass calibration and resolution are verified by the analysis of the instrument performance check solution, decafluorotriphenylphosphine (DFTPP). The instrument performance check

solution, DFTPP, must be injected at the beginning of each 12 hour period of analysis and must meet the following ion abundance criteria:

Mass	Intensity Required (Relative Abundance)
51	30.0 to 80.0% of mass 198
68	<2.0% of mass 69
69	Present
70	<2.0% of mass 69
127	25.0 to 75.0% of mass 198
197	<1.0% of mass 198
198	Base peak, 100% relative abundance
199	5.0 to 9.0% of mass 198
275	10.0 to 30.0% of mass 198
365	>0.75% of mass 198
441	present but less than mass 443
442	40.0 - 110.0% of mass 198
443	15.0 to 24.0% of mass 442

2. Initial and Continuing Calibration -

The initial calibration of the GC/MS system will require a minimum of five concentration levels (20, 50, 80, 120, and 160 total ng/2 µL injection). Eight compounds (2,4-dinitrophenol, 2,4,5-trichlorophenol, 2-nitroaniline, 3-nitroaniline, 4-nitroaniline, 4-nitrophenol, 4,6-dinitro-2-methylphenol, and pentachlorophenol) will require only a four-point initial calibration at 50, 80, 120, and 160 total ng/2 µL injection. The five concentration standards will be analyzed within the same 12 hour period.

The continuing calibration standard will have a concentration of 50 total ng/2 µL injection.

If time remains in the 12-hour time period after meeting the acceptance criteria for the initial calibration, samples may be analyzed. It is not necessary to analyze a continuing calibration standard if the initial calibration standard that is the same concentration as the continuing calibration standard meets the continuing calibration acceptance criteria. If time does not remain, a new injection of DFTPP must meet the abundance criteria given in item 1 followed by a continuing calibration standard.

The relative response factor (RRF) at each concentration level of an initial and/or continuing calibration for each target and surrogate compound must be greater or equal to the compound's minimum acceptable response factor listed in table 1.

The % RSD for each target and surrogate compound from the initial calibration must be less than or equal to the Maximum % RSD listed in Table 1.

The relative response factor percent difference (%Diff) of the initial and continuing calibration for each target and surrogate compound must be less than or equal to the value listed in Table 1.

Up to four compounds may fail the to meet the acceptance criteria listed in Table 1. However, the four compounds must have a minimum RRF $\geq$ 0.010, and the % RSD or % Diff $\leq$ 40.0%.

If a target compound is detected in a sample for which the continuing calibration % Diff >25% for that compound, the sample must be reanalyzed under a continuing calibration with an acceptable % Diff for that compound.

3. Internal Standards -

The minimum required internal standards are:

1,4-dichlorobenzene-d4 naphthalene-d8 acenaphthene-d10,
phenanthrene-d10 chrysene-d12 perylene-d12.

A retention time and response check must be performed on every internal standard for samples analyzed. The final concentration for each internal standard will be 40 total ng/2 µL injection. The retention time (RT) for an internal standard from a sample cannot > ± 30 seconds from the pervious daily calibration. The response area of an internal standard from a sample cannot exceed a factor of 2 (-50% to +100%) from the previous daily calibration.

4. Surrogate Compounds -

The minimum required surrogate compounds are nitrobenzene-d5, 2-fluorobiphenyl, p-terphenyl-d14, phenol-d6, 2-fluorophenol, and 2,4,6-tribromophenol. Required recovery limits follow.

	Water	Soil
Nitrobenzene-d5	35-114	23-120
2-Fluorobiphenyl	43-116	30-115
p-Terphenyl-d14	33-141	18-137
Phenol-d6	10-94	24-113
2-Fluorophenol	21-100	25-121
2,4,6-Tribromophenol	0-123	19-122

5. Method Blanks -

A separate method blank must be extracted and analyzed for each method, matrix, and/or extraction batch. The method blank must be extracted the same day as the field samples it is associated with. Semivolatile organic target compounds must not be present in the blank at a concentration > the estimated quantitation limits (EQLs) with the exception of common phthalate contaminants. The phthalate contamination can be present up to five times the EQL before corrective action is required. Sample results must not be corrected by subtracting any blank value. An instrument blank must be analyzed after any sample that has had a target compound two times the linear range of the instrument (320 ng/ml) to check for carry-over.

6. Samples

Sample (s) must be reanalyzed when one or more of the following occur:

- Acceptance criteria are not met
- Carry over or laboratory contamination is suspected

- A target compound is over the instruments linear range (160 ng/ml). In this case, a dilution is required.

It is important that the lowest possible EQL's are achieved. If the initial analysis of a sample was performed at a dilution and there were no target and/or TICs detected above the EQL, a reanalysis at a higher concentration must occur unless the reanalysis cannot be performed at a level five times the initial diluted analysis. For example, if the initial analysis was at diluted 20% with no target and/TICs detected, a reanalysis without a dilution can be performed. However, if the sample was diluted 25 or 50%, a reanalysis is not necessary and the diluted initial analysis is reported.

When a reanalysis occurs, both sets of data are to be included.

TABLE 1
Acceptance Criteria for Initial and Continuing Calibration of Target and Surrogate Compounds

Target Compound	Minimum RRF	Maximum %RSD	Maximum % Diff
Phenol	0.800	20.5	±25.0
Bis(-2-Chloroethyl)ether	0.700	20.5	±25.0
2-Chlorophenol	0.800	20.5	±25.0
1,3-Dichlorobenzene	0.600	20.5	±25.0
1,4-Dichlorobenzene	0.500	20.5	±25.0
1,2-Dichlorobenzene	0.400	20.5	±25.0
2-Methylphenol	0.700	20.5	±25.0
2,2'-oxybis(1-chloropropane)	0.100	None	None
4-Methylphenol	0.600	20.5	±25.0
N-Nitroso-Di-propylamine	0.500	20.5	±25.0
Hexachloroethane	0.300	20.5	±25.0
Nitrobenzene	0.200	20.5	±25.0
Isophorone	0.400	20.5	±25.0
2-Nitrophenol	0.100	20.5	±25.0
2,4-Dimethylphenol	0.200	20.5	±25.0
Bis(-2-Chloroethoxy)methane	0.300	20.5	±25.0
2,4-Dichlorophenol	0.200	20.5	±25.0
1,2,4-Trichlorobenzene	0.200	20.5	±25.0
Naphthalene	0.700	20.5	±25.0
4-Chloroaniline	0.100	None	None
Hexachlorobutadiene	0.100	None	None
4-Chloro-3-methylphenol	0.200	20.5	±25.0
2-Methylnaphthalene	0.400	20.5	±25.0
Hexachlorocyclopentadiene	0.100	None	None
2,4,6-Trichlorophenol	0.200	20.5	±25.0
2,4,5-Trichlorophenol	0.200	20.5	±25.0

2-Chloronaphthalene	0.800	20.5	±25.0

TABLE 1 - Continue

Acceptance Criteria for Initial and Continuing Calibration of Target and Surrogate Compounds

Target compounds	Minimum RRF	Maximum %RSD	Maximum % Diff
2-Nitroaniline	0.010	None	None
Dimethylphthalate	0.010	None	None
Acenaphthylene	0.900	20.5	±25.0
3-Nitroaniline	0.010	None	None
2,6-Dinitrotoluene	0.200	20.5	±25.0
Acenaphthene	0800	20.5	±25.0
2,4-Dinitrophenol	0.010	None	None
4-Nitrophenol	0.010	None	None
Dibenzofuran	0.800	20.5	±25.0
2,4-Dinitrotoluene	0.200	20.5	±25.0
Diethylphthalate	0.010	None	None
4-Chlorophenyl-phenylether	0.400	20.5	±25.0
Fluorene	0.900	20.5	±25.0
4-Nitroaniline	0.010	None	None
4,6-Dinitro-2-methylphenol	0.010	None	None
N-nitrosodiphenylamine	0.010	None	None
4-Bromophenyl-phenylether	0.100	20.5	±25.0
Hexachlorobenzene	0.100	20.5	±25.0
Pentachlorophenol	0.050	20.5	±25.0
Phenanthrene	0.700	20.5	±25.0
Anthracene	0.700	20.5	±25.0
Di-n-butylphthalate	0.010	None	None
Fluoranthene	0.600	20.5	±25.0
Pyrene	0.600	20.5	±25.0
Butylbenzylphthalate	0.010	None	None

3,3'-Dichlorobenzidine	0.010	None	None

TABLE 1 - Continue

Acceptance Criteria for Initial and Continuing Calibration of Target and Surrogate Compounds

Target compounds	**Minimum RRF**	**Maximum %RSD**	**Maximum % Diff**
Bis(2-ethylhexyl)phthalate	0.010	None	None
Benzo(a)anthracene	0.800	20.5	±25.0
Chrysene	0.700	20.5	±25.0
Di-n-Octylphthalate	0.010	None	None
Benzo(b)fluoranthene	0.710	20.5	±25.0
Benzo(k)fluoranthene	0.700	20.5	±25.0
Benzo(a)pyrene	0.700	20.5	±25.0
Indeno(1,2,3-cd)pyrene	0.500	20.5	±25.0
Dibenzo(a,h)anthracene	0.400	20.5	±25.0
Benzo(g,h,i)perylene	0.500	20.5	±25.0
Aniline	0.010	None	None
Azobenzene	0.010	None	None
Benzyl Alcohol	0.010	None	None
Isophorone	0.010	None	None
N-Nitrosodimethylamine	0.010	None	None
Surrogates			
Nitrobenzene-d5	0.200	20.5	±25.0
2-Fluorobiphenyl	0.700	20.5	±25.0
Terphenyl-d14	0.500	20.5	±25.0
Phenol-d5	0.800	20.5	±25.0
2-Fluorophenol	0.600	20.5	±25.0
2,4,6-Tribromophenol	0.010	None	None
2-Chlorophenol-d4 (Optional)	0.800	20.5	±25.0

1,2-Dichlorobenzene (Optional)	0.050	20.5	±25.0

ORGANICS

SECTION III - SPECIFIC REQUIREMENTS FOR ORGANOCHLORINE PESTICIDES/AROCLORS

I. Overview - Support of operations at Los Alamos National Laboratory, dating back to the Manhattan Project in 1943, has involved the use of pesticides and polychlorinated bi-phenyls (Aroclors). There is reason to believe that some sites/field units involved in DOE's Environmental Restoration Program may contain detectable concentrations of pesticides and/or Aroclors.

The analytical data generated by the subcontractor under this subcontract will be used to determine if there are measurable concentrations of the targeted organochlorine pesticides/Aroclors that will require remediation or long term monitoring.

Sample matrices that the subcontractor may receive (but not limited to) could be water, waste water, soil, sludges, traps, filters, and oils.

The methods cited in this section are methods of the United States Environmental Protection Agency (U.S EPA). Target compounds are those required to meet New Mexico Environment Division or U.S. EPA regulatory requirements.

II. Target compounds/methods citations

ESTIMATED QUANTITATION LIMITS (EQLs) FOR PESTICIDES AND AROCLORS

Analyte	Est. Quantitation limit (µg/L)
Aldrin	*0.05*
α-BHC	*0.05*
β-BHC	*0.05*
δ-BHC	*0.05*
γ-BHC (Lindane)	*0.05*
α-*Chlordane*	*0.05*
γ-*Chlordane*	*0.05*
4,4'-DDD	*0.10*
4,4'-DDE	*0.10*
4,4'-DDT	*0.10*
Dieldrin	*0.10*
Endosulfan I	*0.05*
Endosulfan II	*0.10*
Endosulfan sulfate	*0.10*

Endrin	*0.10*
Endrin Ketone	*0.10*
Endrin Aldehyde	*0.01*
Heptachlor	*0.05*
Heptachlor epoxide	*0.05*
Methoxychlor	*0.50*
Toxaphene	*5.00*
Aroclor-1016	*1.00*
Aroclor-1221	*2.00*
Aroclor-1232	*1.00*
Aroclor-1242	*1.00*
Aroclor-1248	*1.00*
Aroclor-1254	*1.00*
Aroclor-1260	*1.00*

Determination of Estimated Quantitation LIMITS(EQLs) for Various Matrices

Matrix	Factor
Ground Water	1
Low-concentration soil by sonication *	33
High-concentration soil and sludges by sonication*	1000
Non-water miscible waste	10,000

* This factor is based on no GPC clean-up. The factor will vary for soil samples that undergo GPC, based on the GPC equipment used (volume of extract put through GPC). The laboratories should adjust the final volume of the GPC extract to keep make this factor no > 66, if possible.

Methods

The U.S. EPA methods that are options for use are methods SW-8081, **dual column option,** (11/92 or more recent) or the CLP method for pesticides/Aroclors (OLM01.8 or more recent). These methods are based on solvent extraction, concentration, and GC/EC detection and quantitation.

Note: Quality control requirements are specified in Section IV, regardless of the method selected.

Methods 3510, 3520, 3540, 3550 or 3580 can be used for samples submitted for organochlorine pesticide/Aroclor analyses. An overview of these extraction methods and guidance for method selection can be found in method 3500. Gel permeation chromatography (method 3640 or CLP) and/or other cleanup procedures must be performed when the nature of the sample requires it (SW-3600 provides guidance on cleanup usage/procedures).

III. Reporting/Deliverables - There will be a combination of hardcopy and electronic deliverables. Section II.B, Data delivery requirements, of the Statement of Work describes the electronic deliverables for both initial and later term. which is supplied by the Sample Coordinator.

The hardcopy deliverables are as follow and must be in the following order, chronologically arranged, by instrument, and for both columns:

Sample Data

- Copies of pesticide chromatograms labeled with the sample number, volume injected, data and time of analysis, GC column identification (by stationary phase and internal diameter), GC instrument identification, compound names of analytes identified (can be on a printout of retention times if retention times are on the peaks.) This must be provided for both columns.

- GC integration reports or data system printouts

- For pesticides/Aroclors that are confirmed by GC/MS, copies of chromatograms, raw, background-subtracted, and standard reference spectra of target compounds identified are required. For multi-component analytes the mass spectra of 3 major peaks are required.

- Extraction benchsheets including GPC standard data (benchsheet) if GPC was performed.

Standards data

- Chromatograms and data system printouts for all standards. A printout of retention times and corresponding peak areas (or peak heights) must be included, as well as labeling described under sample data (sample number should identify what kind of standard it is).

- Pesticide GPC Calibration Data - UV detector traces showing peaks that correspond to the compounds in the pesticide calibration mix. In addition, start, collect, and dump times must be clearly labeled on the calibration printouts.

- For an interim period while LANL is completing its EDD, the following CLP forms are required: Forms 6E, 6F, 7E, and 7F. See Section II.B of the General Requirements, last paragraph.

Raw QC data

- Blank data, in chronological order, by type of blank.

- Chromatograms and data system printouts for each GC column/instrument used.

- If duplicate matrix spikes are requested, the chromatograms and data system printouts are required.

- Chromatograms and data system printouts for LCSs run in association with LANL samples are required.

QC data must be labeled as the standards data is.

Results must be reported on a dry weight basis.

Note: All hardcopy data must be legible and clearly labeled.

IV. Quality Control (QC) requirements - It is important that the laboratory personnel follow Good Laboratory Practices throughout all operations involved in the analyses of samples. Refer to SW-846 chapter one for general quality control requirements. In addition, quality control requirements for sample extractions and clean-ups for the technique(s) used are found in the relevant SW 846 methods.

Matrix spike and matrix spike duplicate analysis will be requested if needed. A sample will be submitted to be used as the matrix media if matrix spikes are requested and instruction for spiking will be provided..

The QC requirements identified under "Acceptance criteria" must be followed. **Failure to meet the criteria on both columns requires reanalysis of associated samples/blanks under acceptable criteria.**

Acceptance criteria

1. Initial calibration

A minimum of three concentration levels containing the target compounds and surrogates is required. One level at a concentration equal to the estimated quantitation limit (based on the concentration in the final volume specified in the preparation method with not dilution) is required. The other concentration levels should correspond to the working range of the detector.

- The % RSD over the initial calibration range for the calibration factors must be $\leq$ 20% for all target and surrogate compounds.

- For multi-component analytes, a calibration factor for each of 3-5 major peaks is required.

2. Surrogates

The two surrogates to be used are tetrachloro-m-xylene and decachlorobiphenyl. Recovery should be reported as a percentage and must be within 50-160%. Recovery outside of these limits will require re-extraction and reanalysis. If these limits are

exceeded again the data will be accepted, however frequent failures to meet these limits for surrogate recovery will require investigation by the laboratory.

3. Retention time windows

Retention time windows are defined as plus or minus three times the standard deviation from the mean absolute retention times from the concentration levels of the initial calibration. For the multi-component target compounds, choose three to five major peaks and calculate the mean and standard deviation of those peaks. Retention time windows for target and surrogate compounds are to be determined for both the primary and secondary columns.

The retention times will be verified by the calibration verification standard.

4. Daily calibration (calibration verification)

A daily calibration standard will be analyzed prior to sample analysis and throughout the analytical run at a frequency of one per 20 samples. The calibration factor for each target compound must not exceed ± 15% difference when compared to the initial calibration.

5. Blanks

Method blanks must be analyzed along with every analytical run for each matrix and method used. Contamination must not exceed the EQL.

6. Breakdown criteria

The breakdown of either 4,4'-DDT or endrin cannot exceed 20%.

SECTION III - SPECIFIC REQUIREMENTS FOR HIGH EXPLOSIVES ANALYSES

I. Overview - Because of the history of weapons design at Los Alamos National Laboratory, dating back to the Manhattan Project in 1943, there is reason to believe that some sites/field units involved in DOE's Environmental Restoration Program may contain detectable concentrations of high explosives that may have been tested for use as triggering devices during research and development activities for nuclear weapons.

The analytical data generated by the subcontractor under this RFP subcontract will be used to determine if there are measurable concentrations of the targeted explosives compounds that will require remediation.

Sample matrices that the subcontractor may receive (but not limited to) could be water, waste water, soil, sludges, filters, and oils.

The methods that are being cited in this section are those used by either the Environmental Protection Agency (EPA) or the United States Army Environmental Center (AEC) - formerly, the United States Army Toxic and Hazardous Materials Agency (USATHAMA), as are the targeted analytes for these analyses. The methods generally consist of either extraction (liquid or solid phase) of the analytes from the sample matrix or filtering of the sample (water) followed by reversed phase HPLC and UV detection. Nitrocellulose is determined by measurement of nitrate/nitrite, produced by hydrolysis with sodium hydroxide, and measured using ion chromatography. Quantitation of the analytes involves the relationship of the response of the analyte in a sample to a response factor generated using standards.

II. Target compounds/methods citations

The following group of target analytes for high explosives will be referred to as the "8330" analytes because theyare the analytes that are covered under method 8330 in EPA's SW-846.

	"8330 ANALYTES"
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	(HMX)
Hexahydro-1,3,5-trinitro-1,3,5-triazine	(RDX)
1,3,5-Trinitrobenzene	(1,3,5-TNB)
1,3-Dinitrobenzene	(1,3-DNB)
Methyl-2,4,6-trinitrophenylnitramine	(Tetryl)
Nitrobenzene	(NB)
2,4,6-Trinitrotoluene	(2,4,6-TNT)
4-Amino-2,6-dinitrotoluene	(4-Am-DNT)
2-Amino -4,6-dinitrotoluene	(2-AM-DNT)
2,4-Dinitrotoluene	(2,4-DNT)
2,6-Dinitrotoluene	(2,6-DNT)
2-Nitrotoluene	(2-NT)
3-Nitrotoluene	(3-NT)
4-Nitrotoluene	(4-NT)

Holding times for the samples for "8330" analytes are (1) for water samples, extraction must occur within 7 days of sample collection, (2) for soil samples, extraction must occur within 14 days of sample collection, and (3) analysis must occur within 40 days of extraction for both soil and water

samples (number 3 does not, in any way, release the subcontractor from the data turnaround time obligation.)

Methods options for these "8330 analytes" follow. These alternate methods - broken out by soil and water, provide the "estimated quantitation limits" (EQLs) that the subcontractor is required to meet, whether using method SW-8330 and SW-8331 or the USATHAMA methods.

Analytes in Soil	Estimated Quantitation Limits (EQLs) (mg/kg)
HMX	2.2
RDX	1.0
135TNB	0.25
13DNB	0.25
TETRYL	0.65
NB	0.26
246TNT	0.25
2ADNT	—
4ADNT	—
26DNT	0.26
24DNT	0.25
2NT	0.25
4NT	0.25
3NT	0.25

Method Options that can be used:

SW-8330 (11/92) or

USATHAMA AUGUST 1989 REVERSED-PHASE METHOD FOR THE DETERMINATION OF EXPLOSIVE RESIDUES IN SOIL

Analytes in water	EQLs - micrograms/L	
	Low	High
RDX * #	0.84	14.0
135TNB * #	0.26	7.3
13DNB * #	0.11	4.0
NB * #	—	6.4
246TNT * #	0.11	6.9
24DNT * #	0.02	5.7
26DNT * #	0.31	9.4
2ADNT *	0.035	—
4ADNT *	0.06	—
HMX #	—	13.0
TETRYL #	—	20.0
2NT	—	12.0
3NT	—	7.9
4NT *	—	8.5

Where there are blanks above, the EQLs have not been determined. Assume an EQL of 1.0 micrograms/L for the low waters and an EQL of 10 micrograms/L for the high waters where values are missing. As more information is available, it will be provided.

Method options that can be used:

SW-8330 (11/92) or

- USATHAMA 1990 IMPROVED SALTING-OUT SOLVENT EXTRACTION METHOD FOR DETERMINATION OF LOW LEVELS OF NITROAROMATICS AND NITRAMINES IN GROUND WATER

- USATHAMA 6/30/88 METHOD NUMBER UW14, DETERMINATION OF EXPLOSIVES IN WATER BY HIGH PRESSURE LIQUID CHROMATOGRAPHY

Analytes in water samples Assume EQLs of 25 micrograms/L

Nitroglycerine (NG)
Pentaerythritol tetranitrate (PETN)

Method

USATHAMA AUGUST 1989 REVERSED-PHASE HPLC METHOD FOR THE DETERMINATION OF NG AND PETN IN WATER

Analytes in soil samples Assume EQLs of 0.50 mg/kg

Nitroglycerine (NG)
Pentaerythritol tetranitrate (PETN)

Method

USATHAMA AUGUST 1989 REVERSED-PHASE METHOD FOR THE DETERMINATION OF NG AND PETN IN SOIL

Analyte in water Assume EQL of 5.0 micrograms/L

Nitroguanidine

Method

USATHAMA AUGUST 1989 REVERSED-PHASE METHOD FOR THE DETERMINATION OF NITROGUANIDINE IN WATER

Analyte in soil Assume EQL of 0.51 mg/kg

Nitroguanidine

Method

USATHAMA AUGUST 1989 REVERSED-PHASE METHOD FOR THE DETERMINATION OF NITROGUANIDINE IN SOIL

Analyte in water Assume EQL of 6.11 micrograms/L

Tetrazene

Method options that can be used:

SW-8331 or

USATHAMA REVERSE-PHASE HPLC METHOD FOR THE DETERMINATION OF TETRAZENE IN WATER (Holding time is stated as "samples should be processed as soon as possible after receipt, preferably within a day.")

Analyte in soil Assume EQL of 1.3 mg/kg

Tetrazene

Method options that can be used:

SW-8331 or

USATHAMA REVERSE-PHASE HPLC METHOD FOR THE DETERMINATION OF TETRAZENE IN SOIL (Holding time is stated as "samples should be processed as soon as possible after receipt, preferably within a day.")

Analyte in water

Nitrocelluose Assume EQL of 70.0 micrograms/L

Method

USATHAMA METHOD FOR THE DETERMINATION OF NITROCELLUOSE IN WATER

The noted assumptions, above, are based on the USATHAMA lower limit of the linear concentration range.

Holding times for the non-"8330" analytes are the same as for the "8330" analytes. Note that USATHAMA recommends that samples for tetrazene analyses be "processed as soon as possible after receipt, preferably within a day." The subcontractor should take this into consideration when analyzing samples for tetrazene.

Note: Quality control requirements are specified in Section V, *regardless* of the method selected.

III. Reporting/Deliverables - There will be a combination of hardcopy and electronic deliverables Section II.B, Data delivery requirements, of the Statement of Work, describes the electronic deliverables for both initial and later term.

The hardcopy deliverables are as follow and must be in the following order, arranged chronologically, by instrument:

Samples data

- Copies of HPLC chromatograms for each sample (for each column), which are labeled with:
  - sample number
  - volume injected
  - date and time of analysis
  - HPLC column identification
  - HPLC instrument identification
  - names of compounds identified, with retention times, areas, peak heights, and concentrations found (this may be on a summary sheet, if retention times are on the peaks.
- Extraction work/bench sheets.

Standards data

- Copies of HPLC chromatograms as described under "samples data" for all standards

QC data

- Chromatograms of blanks labeled as described under samples data.
- Provide chromatograms of laboratory control samples and matrix spikes (if requested) labeled as described under samples data.

Results must be reported on a dry weight basis.

Note: All data must be legible and properly labelled.

IV. Quality Control (QC) requirements - It is important that the laboratory personnel follow Good Laboratory Practices throughout all operations involved in the analyses of samples for high explosives and that they maintain an internal QC program that is relevant to the analyses under consideration -in this case, high explosives. In addition, it is required to follow the relevant general QC procedures as spelled out in Chapter 1 of SW-846.

Matrix spike and matrix spike duplicate analysis may be requested. A sample will be submitted to be used as the matrix media if matrix spikes are requested and spiking directions will be provided by the Sample Coordinator.

The QC requirements identified under "Acceptance criteria" must be followed. **Failure to meet the criteria requires reanalysis of associated samples/blanks under acceptable criteria.**

Acceptance criteria

1. Initial calibration - external

A minimum of 3 concentrations of the target analytes and surrogate(s), with one of the standards at a concentration equal to the estimated quantitation limit is required. Other concentrations should define the expected range of concentrations in the samples. % RSD must be <20 % over the working calibration range. Either a calibration curve or a single response factor may be used.

2. Retention time windows

The retention time windows for all analytes and surrogates for each HPLC column is established from the initial calibration by the following method:

a. Make three injections of all standards within a 72-hour period.
b. Calculate the mean and standard deviation (σ) of the retention times of each analyte and surrogate.
c. Establish the retention time window as $\pm$ 3 σ from the mean retention time.

Mean retention times of all analytes and surrogates from three injections of the daily standard must fall within the retention time window established by the initial calibration or a new initial calibration and establishment of a new retention time window must be done before running the samples.

If the daily standard retention times are acceptable, the mean of the retention times is used as the midpoint of the retention time window for that day. The width of the window is the $\pm 3\ \sigma$ found in the initial calibration.

All subsequent daily standards at midpoint and end of run must fall within the daily retention time window. If any analyte of any daily calibration fails to fall within the daily retention time window, a new initial calibration must be performed.

3. Daily calibration and response factors

A daily calibration standard is prepared at a concentration midway between the concentrations of the initial standards. This standard is run before the samples to check the condition of the HPLC. It is also run after each group of ten samples, and at the end of the run to ensure system stability throughout the analysis of the samples. Response factors for each analyte must be obtained from the peak height or area and compared with the mean response factors obtained in the initial calibration. The response factor for the daily calibrations must agree within ±15% of the response factor of the initial calibration (±10% for nitroguanidine and tetrazene). Corrective action to the analytical system or a new initial calibration must be performed if these criteria are not met.

4. Laboratory control sample

Complete system performance will be monitored by using a laboratory control sample. A known amount of 7 to 10 of the analytes plus surrogate equal to 10x EQL of that established for the analytes is added to the matrix to be analyzed. For water samples, it is added to organic free reagent water; and for soil samples, it is added to standard soil.

The control samples are carried through the laboratory procedure and analyzed as samples.

A laboratory control sample is run with each batch of samples and the results are compared with the known amount. Advisory limits for recovery are 60-120 % for water and soil samples. As information is gathered, these windows will be revised

.5. Blank sample

A blank is prepared by adding a known amount of the surrogate to either reagent water or clean sand. The blank sample is run with each batch of sample and the recovery of the surrogate is found. If target analytes are found at greater than EQL, contamination must be corrected before further analyses are performed.

6. Surrogate

Acceptable surrogates are compounds similar to the target analytes but not present in the samples. Two surrogates in use are 3,4-dinitrotoluene (required) and 2-methyl-4-nitroaniline (MNA) (optional). The surrogate(s) is added to samples, blanks, and LCSs and the complete laboratory procedure is carried out. Surrogate recovery is reported for all samples, blanks, and LCSs. Windows will be developed for acceptable recovery as data is collected.

SECTION III - SPECIFIC REQUIREMENTS FOR RADIOISOTOPE ANALYSES

I. **Overview** - Because of the history of weapons design at Los Alamos National Laboratory, dating back to the Manhattan Project in 1943, some sites / field units involved in DOE's Environmental Restoration Program will contain detectable concentrations of radioactive material that have been used during research and development activities for nuclear weapons.

The analytical data generated by the subcontractor under this subcontract will be used to determine if there are measurable concentrations of the targeted isotopes that will require remediation.

Sample matrices that the subcontractor may receive, include (but are not limited to) water, waste water, soil, sludge, filters, and oils.

II. **Target analytes** - Table III.B.1 contains the analyte target list for radiochemical analyses.

The methods utilized by the lab for the analyses below must be submitted to LANL for approval. If following approval of a lab's method, LANL observes performance problems with the approved method the lab will be required to take the necessary actions to resolve the problems. Revisions to previously approved methods shall be supplied, along with any performance data generated by the lab as part of their own approval process, to LANL on a timely basis (approximately 2 weeks) following their issuance.

III. Reporting/deliverables

Hardcopy

- Sample preparation worksheets
- Software outputs for instruments on a sample-by-sample basis
- NIST traceability for standards
- Calibration information.

LANL may request additional hard copy data deliverables based on the subcontractor's specific method/equipment.

IV. Quality Control (QC) requirements

1. Good Laboratory Practices

It is important that the laboratory personnel follow Good Laboratory Practices throughout all operations involved in the analyses of samples for radioisotopes and that they maintain an internal QC program that is relevant to the analyses under consideration.

Laboratories should observe the guidance of the Good Automated Laboratory Practices (GALP), EPA December 28, 1990 draft, document in their handling electronic files representing sample raw data, sample processed data, and instrument calibration and configuration information

2. Control Charts

For all analytes of interest, the analytical laboratory shall maintain internal control charts for lab control standard samples of both water and soil matrices. The analytical laboratory shall initiate these control charts prior to beginning LANL work and update them as data becomes available. The lab will be expected to use professional judgement in use of resulting statistical control information.

3. Instrument Performance Verification and Calibration

Instrument performance verification and calibration shall be performed at the frequencies indicated in table III.E.3.1. The respective control charts shall be updated as data becomes available or daily, whichever is greater.

The data resulting from performance verification measurements shall be charted using the guidance of ANSI N42.2, *Measurement Quality Assurance for Radioassay Laboratories*, (section A.5.2.2) and *Manual on Presentation of Data and Control Chart Analysis* (ASTM STP 15D, Part 3, section 33 (individuals), ASTM 1976). Th acceptance criteria for statistical control in table III.E.2 shall be observed. In the circumstance that one or more of these acceptance criteria are not met then corrective action up to and including re-calibration shall be done and documented. The corrective action process to be followed by the lab should be documented in an SOP.

The indicated calibration frequencies assume no change in instrument settings (e.g. bias voltage) or components (e.g. gas-proportional counter window or laser dye). If such settings or components are changed the lab will conduct the necessary calibrations.

4. Acceptance Criteria

Failure to meet the tracer or carrier recovery criteria for a LANL sample requires re-analysis of that sample at least once. A subsequent failure shall be indicated in the case narrative. Failure to meet tracer and carrier recovery criteria for the reagent blank or lab control standard requires re-analysis of all associated LANL samples under acceptable criteria. Failure to meet the lab control standard or method blank criteria requires re-analysis of all associated LANL samples under acceptable criteria. If re-analysis for any of the above reasons is prevented by exhaustion of the supplied sample this shall be indicated in the case narrative

a Tracer recoveries

Tracer recoveries for alpha emitters shall be greater than or equal to 30% but less than or equal to 110% Tracer recoveries for other than alpha emitters shall be greater than or equal to 40% but less than or equal to 110%.

b Carrier recoveries

Carrier recoveries shall be equal to or greater than 40% but less than or equal to 110%

c. Method blanks

Method blanks shall be prepared along with samples in the SDG/RN. These shall include all preparation steps and all reagents for each analysis and for the matrix (i.e. soil, water) which matches that of the associated SDG/RN. Method blank results shall not exceed the EQL in table III.B.1 LANL shall consider the aliquot size of the associated samples in evaluating the acceptability of the reported method blank result.

d. Lab Control Standard (LCS)

Standards traceable to NIST in a matrix comparable to the associated samples shall be run at a frequency of one per SDG/RN. Analyte recoveries must be ± 25% of the certified values. The LCS need not contain all analytes reportable for that analysis.

The activity of the LCS should be in the range of five to fifty times the respective EQL. Where the contractual EQL is 0.01 pCi/g the activity of the LCS should be in the range of fifty to two hundred times the respective EQL.

e. Matrix spikes (MS)

Those analysis where matrix spikes are required include: strontium-90 when a strontium-85 tracer is not used, tritium by liquid scintillation, total uranium by KPA, mass spectroscopy techniques, radium-226 by other than tracer techniques, radium-228, thorium-234, and lead-210. The matrix spike activity shall be added prior to the beginning of sample digestion or other wet chemistry.

The activity of the matrix spike should be in the range of five to fifty times the respective EQL. Where the contractual EQL is 0.01 pCi/g the activity of the matrix spike should be in the range of fifty to two hundred times the respective EQL. The MS need not contain all analytes reportable for that analysis.

Matrix spikes, where required, shall be run at a rate of 1 per 20 samples or per LANL SDG/RN, whichever is greater. Matrix spike recoveries should be within 25% of the expected value. The lab will be expected to note in the case narrative any matrix spike recovery outside of this limit and to use professional judgement in deciding if re-analysis of associated LANL samples is necessary.

f. Duplicates

At least one sample in each SDG/RN shall be processed in duplicate and reported. There are no required acceptance criteria for these duplicate results. Where there is insufficient sample to allow a duplicate analysis to be done the situation shall be noted in the case narrative.

In the situation where only one sample is available for both the duplicate and matrix spike analysis and their is insufficient mass/volume available, the duplicate analysis shall take precedence.

V. Technique specific requirements

1. General

a. The total propagated uncertainty (TPU) associated with reported results shall be one sigma, 68% confidence interval. TPU shall include reasonable and appropriate systematic uncertainties associated with analysis in addition to the uncertainty from counting statistics. Labs are referred to *Data Reduction and Error Analysis for the Physical Sciences* (P.R. Bevington; McGraw-Hill, 1969; 56-64) for the propagation of counting and systematic uncertainties.

b. Subtraction of method blank results from sample results shall not be done.

In the analysis of tritium, we would consider the vial counted for purposes of background subtraction as separate from those counted as a result of the required method blank preparations.

c. Each lab shall use NIST traceable standards for calibration, tracer, and LCS purposes. Dilutions of NIST traceable standards shall be documented in a controlled notebook or binder. When a NIST traceable standard is unavailable for a given analysis, the use of standards that are not NIST traceable shall be included in the report narrative with historical background information and/or the basis for the known value.

d. The certificate specified expiration date of standards or reference materials should be observed. However, where the vendor has provided supplementary documentation of a longer useful life of the standard or reference material this may be used. This supplemental documentation shall be retained with the certificate.

e The following general equation for MDA (minimum detectable activity) shall be used unless otherwise noted herein or approved by LANL:

$$MDA = [4.65\ (BKG)^{0.5} + 2.71]/[2.22 * EFF * V * T_s * Y]$$

Where BKG is the total background counts, Ts the sample count duration, EFF is the fractional detector efficiency, V is the volume or unit weight, and Y is the fractional chemical recovery obtained for the tracer or carrier. Other terms as may be required, e.g. gamma abundance, can be used in the denominator.

Those labs wishing to use the decision level principal of ANSI N42.2, Measurement Quality Assurance for Radioassay Laboratories, are welcome to do so. However, we would expect the inclusion of information in the data package regarding the "nominal values of a number of parameters (background count rate, count time, estimated interferences, chemical recoveries, decay times, etc.)" used to arrive at the stated decision level. The source of the nominal values shall be addressed in an applicable SOP

The determined MDA or ANSI N42.2 decision level shall be less than the respective EQL indicated herein. Technical difficulties, e.g. insufficient sample, that prevent meeting the respective EQL requirement shall be documented in the case narrative.

f. Counters shall not be reserved for a particular type (e.g. method blanks or other QC sample) of sample. Counters may be reserved for specific nuclides.

g. Where a lab's verification of the activity of a NIST traceable standard indicates a noticeable deviation from the certified value, the lab should consult with the provider about the problem. However, except where permitted here-in the lab shall not use a value other than the decay corrected certified value.

h. Results for soil matrices shall be reported on a dry weight basis for all parameters except tritium.

i. Unless herein specified, the soil aliquots used in wet chemistry techniques shall be subjected to a total digestion or fusion prior to analysis. No other homogenization or size exclusion steps are required.

2. Alpha spectroscopy

a. The presence, evaluation, and explanation of any unusual peaks, greater than 10% of the tracer peak, beyond those of the tracer and expected analyte peaks should be included in the report narrative.

b. The area counts of an analyte or tracer region should be determined by integration of counts in defined ROI unless the number of counts in the respective region exceed 25. In the situation where the area counts of a region exceed 25 peak fitting algorithms may be used.

c. Labs using uranium-232 as a tracer in sequential isotopic uranium/thorium determinations should take steps to minimize the contribution of thorium-228, from uranium-232 decay, in their reported isotopic thorium results.

d. Each alpha spec LCS should contain sufficient activity, as required herein, of each isotope to be quantified to yield as useful result. The exception to this will be uranium-235 in isotopic uranium analyses.

e. In the case of analysis for isotopic uranium only the uranium-234 and uranium-238 will be subject to the LCS and MS recovery criteria. However, the amount of uranium-235 in the LCS and MS spike as well as the uranium-235 results for the LCS and MS shall still be reported. If the uranium spike solution consists of enriched uranium with elevated levels of uranium-235 then the LCS and MS recovery criteria will also apply to uranium-235.

3. Liquid scintillation for tritium

a. The analytical batch LCS sample shall be positioned ahead of all the sample vials in the count sequence. The final two vials of the count sequence shall be an instrument check standard and instrument background check vial. These tailing instrument control check vials may be separately/previously prepared vials. The tailing instrument background vial result needs not be used in any result calculations. Information on the known activity and reference time of the tailing instrument check standard vial should be included in the package.

b. If the quench value of any reported sample lies outside the instrument quench curve range, this shall be noted in the case narrative.

c. The duration of the dark/temperature adaption procedure shall be addressed in the lab's SOP or analytical documentation.

d. The same amount of sample and cocktail shall be used for all samples within a batch. The amounts of each should be indicated on a vial preparation log sheet. This sheet should also indicate the brand name of the cocktail or the composition if not a name brand.

e. The LSC counting protocol should be set up to report the count rate outside the tritium counting region or demonstrate through spectra the lack of significant counts beyond the tritium region. This information shall be provided to LANL.

f. For tritium in soil analyses, result shall be reported in pCi per volume of extracted water. Volume units of mL or L are both satisfactory, i.e. pCi/mL or pCi/L. The percent moisture of the sample shall also be determined for any soil matrix for which tritium is also requested.

g. Soil samples for tritium analysis should be preferably frozen but shall at least be refrigerated prior to analysis.

h. Labs should attempt to use a soil sample size sufficient to, considering the sample soil moisture, allow extraction of the necessary volume of native soil moisture for counting. If the available sample size/soil moisture requires the addition of dead water this added aliquot shall be accounted for in the calculation of the native soil moisture tritium activity.

i. Sample aliquots of aqueous samples shall be distilled prior to analysis.

4. Gamma spectroscopy

a. The isotopes to be quantified and their respective energies, abundances, and half-lifes will be supplied by LANL. These isotopes and the above quantitation information may be updated on a periodic basis.

For those software packages allowing an identification only library, LANL will supply a list of isotopes and energies that should be used in the analysis of LANL samples. These isotopes and energy information may be updated by LANL on a periodic basis.

All isotopes requested by LANL need not be included in the LCS determination.

b. The same calibration files (energy-efficiency curve, detector background, peak shape characteristics, and internal absorption) for a given detector shall be used in the software analysis of all samples within a batch. The lab may use differing channel-energy files as long as the determinations are documented.

c. Where there is sufficient sample, the sample duplicate will be a separately prepared container of sample, not a recount of the same sample container. If there is not sufficient sample, this will be indicated in the report narrative. Where multiple detectors are used in the analysis of LANL samples the sample duplicate will be counted on a detector different from that used for the count of the associated sample.

d. The geometry of a sample shall reflect that of the associated calibration standard. Dilution may be used in those cases where the sample size is less than the smallest available calibrated geometry. Otherwise a calibrated geometry sufficiently small to accommodate the available sample should be used.

e. The report narrative should indicate which analysis software package and version was used to analyze the reported spectra.

f. The lab SOP for the gamma analysis should indicate the software criteria used to analyze the reported spectra. The lab SOP should also provide listings of relevant software macros used in the acquisition/analysis of spectra.

g. For those software packages that allow the user to choose the manner in which the gamma MDA is calculated, the choice should be that of Currie (Lloyd A. Currie, Analytical Chemistry, 40(3), March 1968, 586-93).

h. For decay corrections, the reference time will be the time and date that the sample is collected. Where time of collection is not specified 1200 hours (noon) should be used.

i. The use of europium-154 and -155 sources for calibrations used in the quantitation of LANL samples is not permitted.

j. The efficiency calibration source shall have emissions at the following approximate energies produced by the indicated isotope: 59.5 keV (americium-241), 88 keV (cadmium-109), 122 keV (cobalt-57), 166 keV (cerium-139), 279 keV (mercury-203), 392 keV (tin-113), 662 keV (cesium-

137/barium-137m), 898 keV (yttrium-88), 1173 keV (cobalt-60), 1332 keV (cobalt-60), and 1836 keV (yttrium-88). These calibration sources shall not be used for efficiency, energy, or peak shape calibrations beyond one year since certification of the source. In addition, no isotope in this source will be used beyond five half-lifes since the certification date. Sources beyond this one year period may be used as a LCS source.

k. It shall be acceptable to chart the performance parameters of only americium-241, cesium-137, and cobalt-60 in meeting the requirements of section III.E.2. The parameters that should be monitored include recovery, peak energy, and peak resolution.

l. In making detector background determinations, the lab may use its own judgement in using either an empty cave, a cave with an empty counting geometry, or a cave with an appropriate de-ionized/distilled water filled counting geometry.

m. If the lab's detector is incapable of detecting the emissions of requested isotopes with energies below that of americium-241 these need not be reported. For those lab's that are capable of detecting emissions of these isotopes the lack of complete calibration of detection efficiency at these energies is acknowledged.

n. Where the setting is allowed by the lab's software, all counts should have an abundance limit set at 75%.

o. Vendor supplied training in the specific software being used for gamma spectral analysis by the respective analyst(s) is strongly encouraged.

p. The soil sample aliquot that is counted shall have been previously dried.

5. Gross alpha/beta

a. A planchet residue coverage density limitation of 5 mg/cm^2 for alpha/beta determinations and 10 mg/cm^2 for beta only determinations should be observed when using a 2-inch planchet. However a planchet residue coverage density of up to 10 mg/cm^2 for alpha/beta determinations may be used if an additional LCS sample with a planchet residue coverage density of 7.5 to 10 mg/cm^2 is run with each SDG/RN.

b. Prepared planchets should not be flamed if they are maintained in a desiccated environment prior to counting. If flaming is necessary then this should be included in the case narrative.

c. The acidity of aqueous samples should be checked before analysis and pH < 2 confirmed. Samples whose pH does not meet this criteria should be acidified and analysis held for 16 hours. LANL SMO should be notified which samples required acidification by the lab in this manner and the report narrative should also indicate this fact.

d. It is permissible for the laboratory to use other than the certified value in assessing the recovery of their LCS and MS recoveries due to the gross nature of this analysis. The approach taken by the lab to derive the "apparent" known value of the LCS and MS spike shall be documented in an SOP and referenced in the data package along with certified known value.

6. Uranium by KPA (**Note that this is not an analysis under the routine analyses categories, but is a commonly-requested non-routine analysis.**)

a. Individual measurements shall have a minimum life-time of 200 µsec. and an R^2 of greater than 0.97. If sample dilution does not allow this criterion to be met the lab should note the problem in the case narrative. This criterion is not applicable if the concentration is below the EQL indicated in table III.B.1.

b. LANL reserves the right to require the use of the method of standard additions (MSA) in the analysis of samples by KPA.

c. Total digestion or fusion is not required for KPA analysis of soil samples.

7. Elemental uranium by ICP-MS

a. The lab's procedure should be based on EPA method 6020 with uranium specific quantitation guidance from EPA EMSL-CIN method 200.8, "Determination of Trace Elements in Waters and Wastes by ICP-MS". Any deviations from EPA method 6020 in the lab's procedure shall be submitted for approval as provided for in section III.B.

8. Strontium-90

a. Where the strontium-90 activity of a sample is expected to exceed 5 pCi/g the sample should be re-counted a few days hence so as to observe an expected increase due to additional yttrium-90 ingrowth. It is only necessary to document this count and the results in the supplied data package. A complete calculation of an additional final result is not necessary.

9. Gross gamma

a. It shall be permissible for the laboratory to use other than the certified value in assessing the recovery of their LCS and MS recoveries due to the gross nature of this analysis. The approach taken by the lab to derive the "apparent" known value of the LCS and MS spike shall be documented in an SOP and referenced in the data package along with certified known value.

10. ICP-MS/ICP-MS-FIA (Non-routine analytical service frequently requested)

a. Total propagated uncertainty values should not be reported for results determined by IPC-MS or ICP-MS-FIA techniques.

b. Minimum detectable activities/concentrations for ICP-MS/ICP-MS-FIA techniques shall be determined based on standard practices.

Table III.B.1 Target analyte EQL[1] by matrix; pCi/g or pCi/L unless indicated			
Analyte	Soil	Water	Technique[2]
Gross alpha/beta	10.0	3.0	gas-proportional
Gross alpha/beta	10.0	NA	liquid scintillation
Strontium-90[3]	2.0	5.0	gas-proportional
Americium-241	0.1	0.1	alpha spectroscopy
Plutonium-238, -239	0.1	0.1	alpha spectroscopy
Thorium-228, -230, -232	0.1	0.1	alpha spectroscopy
Thorium-230, -232	0.1	0.1	ICP-MS-FIA (commonly-requested non-routine analysis)
Uranium-234, -235, -238	0.1	0.1	alpha spectroscopy
Uranium-234, -235, -238	0.1	0.1	ICP-MS-FIA (commonly-requested non-routine analysis)
Tritium	300 pCi/L	300	liquid scintillation
multiple isotopes (Table III.F.4)	Am-241: 1 Cs-137: 1	Am-241: 20 Cs-137: 20	gamma spectroscopy
Gross gamma	2.0	100	NaI(Tl) or HPGE detection
Total uranium	0.5 µg/g	1 µg/L	KPA[4] (commonly-requested non-routine analysis)
Total uranium	0.5 µg/g	1 µg/L	ICP-MS (commonly-requested non-routine analysis)
Radium-226	1.0	1.0	assorted
Radium-228	0.5	0.5	assorted
Thorium-234	1.0	20	assorted

Table III.B.1 Target analyte EQL[1] by matrix; pCi/g or pCi/L unless indicated			
Analyte	Soil	Water	Technique[2]
Lead-210	2.0	5.0	assorted

1) Estimated Quantitation Limit (EQL)
2) The Los Alamos National Laboratory methods for these analytes are contained in LA-10300-M, "Health and Environmental Chemistry: Analytical Techniques, Data Management, and Quality Assurance".
3) It may be presumed that strontium-89 is not present.
4) Kinetic Phosphorescence Analysis, also referred to as pulsed-laser phosphorimetry (ASTM D 5174-91) or kinetic laser phosphorescence

Table III.E.2. Statistical Control Acceptance Criteria	
Rule No.	Rule
1	1 point above 3 sigma
2	2 of 3 points above 2 sigma
3	4 of 5 points above 1 sigma
4	8 consecutive points above center line
5	1 point below -3 sigma
6	2 of 3 points below -2 sigma
7	4 of 5 points below -1 sigma
8	8 consecutive points below center line
9	15 points inside ±1 sigma
10	8 points outside ±1 sigma
from ASTM C1210-91, table 1	

Table III.E.3.1 Instrument Verification and Calibration Frequency		
Instrument and/or Technique Calibration Type	ication Frequency	ration Frequency
ectroscopy		
energy-channel	weekly	monthly
detector background	weekly	monthly
detector efficiency	weekly	semi-annual
pectroscopy		
channel-energy	weekly	annual
detector background	weekly	monthly
detector efficiency	weekly	annual
peak shape characteristics	weekly	annual
ortional Counting		
mass-attenuation		annual
detector efficiency	aily before use	annual
detector background	y before counting	weekly
cross-talk	quarterly	annual
platenu voltage	quarterly	annual
	per EPA 6020	
instrument calibration	aily before use	weekly
instrument background		aily before use
intillation		
uench curve/detector efficiency	aily before use	semi-annual
counter background	each count batch	

Table III.E.3.1 Instrument Verification and Calibration Frequency		
Instrument and/or Technique Calibration Type	ication Frequency	ration Frequency
ma Spectroscopy		
detector efficiency	aily before use	semi-annual
detector background	aily before use	weekly

Table III.F.4 Gamma Spectroscopy Analyte Requirements			
Nuclide symbol	Nuclide name	Nuclide symbol	Nuclide Name
Ac-228	actinium-228	Pa-231	protactinium-231
Am-241	americium-241	Pa-233	protactinium-233
Ann Rad	annihilation radiation	Pa-234m	protactinium-234m
Ba-140	barium-140	Pb-210	lead-210
Bi-211	bismuth-211	Pb-211	lead-211
Bi-212	bismuth-212	Pb-212	lead-212
Bi-214	bismuth-214	Pb-214	lead-214
Cd-109	cadmium-109	Ra-223	radium-223
Ce-139	cerium-139	Ra-224	radium-224
Ce-144	cerium-144	Ra-226	radium-226
Co-57	cobalt-57	Ru-106	ruthenium-106
Co-60	cobalt-60	Rn-219	radon-219
Cs-134	cesium-134	Se-75	selenium-75
Cs-137	cesium-137	Sn-113	tin-113
Eu-152	europium-152	Sr-85	strontium-85
Hg-203	mercury-203	Th-227	thorium-227
I-129	iodine-129	Th-234	thorium-234
K-40	potassium-40	Tl-208	thallium-208
La-140	lanthanum-140	U-235	uranium-235
Mn-54	manganese-54	Y-88	yttrium-88
Na-22	sodium-22	Zn-65	zinc-65
Np-237	neptunium-237		