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CHARACTERIZATION OF EMISSIONS PRODUCED BY THE OPEN BURNING/OPEN DETONATION THERMAL TREATMENT OF COMPLEX MUNITIONS

by

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and

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WEST DESERT TEST CENTER
U. S. ARMY DUGWAY PROVING GROUND
DUGWAY, UTAH 84022-5000

VOLUME 3 - Appendices G - L (Letters of Instruction)

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APPENDIX G. QUALITY ASSURANCE/QUALITY CONTROL

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A QA PROJECT PLAN FOR:
DUGWAY PROVING GROUND BANGBOX STUDY
STRATEGIC ENVIRONMENTAL RESEARCH AND DEVELOPMENT PROGRAM

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M	SAFETY ANNEX FOR OPEN BURNING/OPEN DETONATION (OB/OD) TESTING WITHIN THE COMBUSTION PRODUCTS TEST FACILITY

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1.0 Program Description

A considerable quantity of obsolete and/or unsafe propellant, explosives and pyrotechnics (PEP) are stockpiled and must be disposed of in a safe, effective, and efficient manner. The most appropriate way to dispose of most of these materials is by open burning (OB) or open detonation (OD). Of environmental concern, however, is the possible pollution of the air, soil, and water during such operations.

Previous studies (OB/OD Phases "A", "B", and "C") involving the open burning and open detonation of sizable quantities of these materials, up to 10,000 pounds in a single test, have demonstrated the acceptability of such methods. The concentrations of identified pollutants generated during such tests, measured using EPA-validated methods, were very low -- much less than current environmental standards.

In addition, a number of studies involving the detonation/burning of much smaller quantities, up to several pounds, within a closed hemispherical chamber, called a BangBox™ (BB), demonstrated comparable levels of concentrations of pollutants in the air from the BB tests with those generated in open air. Because these demonstrations have proven the validity of BB test results, the EPA (Region VIII) has reviewed the results and accepted BB testing as an acceptable method for demonstrating the safe destruction of

these PEP items, and has deemed these tests as acceptable for supporting RCRA Part B applications. Moreover, the costs of time, materials, and labor of testing in the BB are much less than of open air tests. As a result, two series of tests, consisting of ten munition items each, from the Air Combat Command (ACC) operations were carefully selected (as representative of various groups of items in the inventory) for testing as Series ACC-1 and ACC-2 in well-controlled test series in the specially-designed and constructed BB facility at DPG. These tests have been reported as successful for their intended purposes. In addition, a number of special tests to determine the products of combustion from a chlorinated propellants have been successfully conducted.

The sampling and analytical methods used in previous open-air and BB tests have demonstrated the ability to measure volatile organic compounds (VOCs) at parts per trillion, and semi-volatile organic compounds (SVOCs) at nanogram levels.

This program brings together the expertise of scientists from ECO, L.C. (ECO), Mountain States Analytical Incorporated (MSAI), Dugway Proving Ground (DPG), Oregon Graduate Institute (OGI), Radian Corporation (Radian), Sunset Laboratories (SUN), and MHW Laboratory (MHW). The various organizations and laboratories have prepared Letters of Instruction (LOIs) which detail their sample handling, sample preservation, and analytical operations. Instructions for

preparing LOIs have been previously sent out to the laboratories for their guidance. (Appendix A contains a copy of Instructions for Preparing LOIs). Technical and QA support from the EPA¹ has been provided by the Research Monitoring and Evaluation Branch (RMEB) of that Laboratory.

Complete copies of the Test Design Plan (TDP) (Ref. 8) may be obtained from Test Director, West Desert Test Center, STEDP-WD-P-E, (J. L. [Casey] Wilcox), Dugway, Utah 84022.

2.0 Test Matrix

A test matrix has been approved by the BB Technical Steering Committee, the expert panel of scientists and engineers that oversee and advise the DPG Test Director. An abbreviated test matrix is shown in Table 1.

¹ Wherever EPA appears hereinafter, it will refer to: U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Research Monitoring and Evaluation Branch, Research Triangle Park, NC 27711.

Table 1. Test Matrix for the SERDP

<u>No. Munition/Test Item</u>	<u>Type of Initiation</u>	<u>Maximum NEW* (g)</u>
1. Detonation/Initiation Train	Detonation	227
2. TNT/Ammonium nitrate (Amatol)	Detonation	227
3. TNT/RDX/wax (Composition B)	Detonation	227
4. TNT/RDX/Aluminum/wax	Detonation	227
5. Low-density Tritonal (TNT/Aluminum)	Detonation	227
6. Low-density Tritonal with organics	Detonation	227
7. Low-density Tritonal with water	Detonation	227
8. ARD 446-1 impulse cartridge		
-- gas ignition*	Burn	2270
9. Waste energetic material	Burn	1135
10. Diesel/dunnage	Burn	2270

NEW = Net Explosive Weight per trial

TNT = 2,4,6-Trinitrotoluene

RDX = Cyclotrimethylenetrinitramine

* Due to lack of proper gas ignition equipment, this series was deleted, and in its place a detonation test of Amatol with water was substituted.

The above list of the munitions is in the original planned order for testing.

3.0 Project Organization and Responsibilities

Personnel from the various organizations involved will be assigned commensurate with their responsibilities. Many of the personnel in responsible positions for this study have been directly involved with the OB/OD program in earlier phases -- some since its beginning. This continuity of personnel involvement and prior direct experience provides an added measure of confidence and reliability toward the success of this project.

<u>Organization</u>	<u>Responsibility</u>
Dugway Proving Ground ECO, L.C. Cecil Eckard H. Smith Broadbent Wayne Ursenbach R. C. Rhodes Robert Allen Radian Corporation	Test site coordination Project management and QA Project Manager Chemistry consultant Explosives consultant Consultant for Quality BangBox Field Operations and QC Semi-volatile organic chemical analyses and QC
Oregon Graduate Institute Mountain States Analytical	Canister analyses and QC Gravimetric, semi-volatile organic chemicals, and metals analyses and QC
Sunset Laboratories MHW Laboratory	Inorganic and organic carbon and QC Carbon, Hydrogen, and Nitrogen analyses and QC
EPA, RMEB, RTP, NC	QA support

Other scientific experts may be consulted periodically as the project progresses. As indicated above, each operating organization will be responsible for its internal quality control. EPA/RTP/RMEB in several recent studies, ACC-1 and ACC-2, provided considerable quality assurance support by performing systems audits of field and laboratory operations. The results of these quality systems audits, with few minor deficiencies, were generally commendatory. Further, the results of the performance audit of OGI was excellent. The organizational entities and responsibilities for this SERDP study are identical to those under the ACC-1 and ACC-2 studies. Consequently, because of (1) the excellent audit results for ACC-1 and ACC-2, and (2) the continuity of activities and responsibilities it was deemed appropriate by the DPG Test Director, the ECO Project Manager, and the Quality Assurance Project Officer for this study to rely on the results of these very

recent EPA audits rather than incur the added time and expense of additional audits for this specific study. R. C. Rhodes, Consultant for Quality to ECO, will be responsible for the overall Quality Assurance of the project -- including a review/evaluation of the internal quality control systems of the laboratories.

4.0 QA Objectives for Measuring Data Quality in Terms of Precision, Accuracy, Completeness, and Detection Limits

Table 2 Summarizes the precision, accuracy, completeness, and detection limit goals by task. These data are part of the QC program for the laboratories which are described in more detail in Section 10 and in the laboratory LOIs. The values generated in this table were based on the results from previous BB tests, Field Test Phase "A", Field Test Phase "B", and Field Test Phase "C".

While detailed explanations and calculations for precision, accuracy, completeness, and detection limits are provided in Appendix C, brief definitions are provided below. These definitions are derived from documents obtained from Rhodes (Ref. 5). Other useful information may be found in the EPA Quality Assurance Handbook (Ref. 2) and Taylor (Ref. 7).

Precision is the degree of mutual agreement among individual measurements which is often expressed as a percent Relative Standard Deviation (% RSD). **Accuracy** is the difference between the

Table 2. Precision, accuracy, and detection limit objectives

Method/Analyte	Precision/Method	Accuracy/Method	Detection Limits
Exotic Organics	±0-148 ng/sample duplicate samples (depends on analyte) ^a	± 50%/audit samples, lab standards	0.03-196 ng/mL (depends on analyte)
Semi-Volatiles (filters)	± 0-103 ng/sample duplicate samples (depends on analyte) ^a	± 50%/audit samples, lab standards	0.03-196 ng/mL (depends on analyte)
Metals (filters)	± 10%/duplicate samples	± 20% Al } ± 10% Si } NIST standards ± 3% others } USGS Standards	2-150 ng/cm ² (depends on analyte)
Volatiles (6 Liter canisters)	± .2% - ± 10% /duplicate samples (depends on analyte)	± 15-20%/audit samples, lab standards	1 ppt - 10 ppb (depends on analyte)
Real-time gases (CO, CO ₂ , O ₂ , NO, NO ₂ , NO _x , SO ₂)	± 15%/duplicates	± 15%/audit samples, lab standards	5.0 ppb - 1.2 ppm ^b
Organic and Elemental Carbon	± 4-6 %	± 4-6 %	0.2 - 0.3 µg/cm ² of filter
HCl (by GFC analyzer)	± 0.2 ppm	± 2 %	0.20 ppm
HCl (by Method 26)	6.2 %RSD at 3.9 ppm 3.2 %RSD at 15.3 ppm	zero at < 50 ppm	0.1 µg/ml
^a Analyte	<u>Detonation Filter</u>		
2-Nitronaphthalene	24 (1.4)		
2,4-Dinitrotoluene	46 (0.70)		
2,6-Dinitrotoluene	5.9(0.28)		
2,4,6-Trinitrotoluene	70(2.1)		
Naphthalene			
1,3,5-Trinitrobenzene			
Phenol	2183(103)		
^a All quantities listed as: x ng/sample (standard deviation)			
^b CO ₂ =1.2 ppm, CO=0.1 ppm, O ₂ =5.0 ppb, NO _x =6.0 ppb, NO ₂ =6.0 ppb, NO=6.0 ppb, SO ₂ =2.0 ppb			

average value of a set of measurements of a standard and the reference value of the standard and may be expressed as a percent bias (%B). Completeness is the total number of samples collected and analyzed compared to what should have been collected and analyzed based on the Test Design Plan. Thus, overall completeness could be expressed as two percentages, one for sampling/collections and one for analyses. Or the completeness can be computed for each type of sample and each type of analysis. The idealistic goal or

objective for the field collection of samples if obviously 100%. However, being realistic and considering the fact that the tests for each munition are to be performed in triplicate, a reasonable and attainable goal is considered as 95%. Analytical laboratories generally consider that a goal of 90% or better is reasonable and achievable for the analyses.

Detection limit refers to the lower limit at which a measurement system can detect the presence of a specified analyte. There are many different detection limits depending upon which specific "noise" distribution is considered. The lowest possible detection limits are the instrument detection limits, based only on the fluctuations of the instrument noise. The largest "noise" distribution in this series of tests, for a given munition, is the variation among the three replicate detonations of that munition. Instrument detection limits are generally given as the signal-to-noise ratio, usually 3:1. Various rules have been defined concerning the reporting of values near the detection limits. See "Guidelines for Data Acquisition and Data Quality Evaluation in Environmental Chemistry", Analytical Chemistry (Ref. 1) and "Too Much Ado About Next to Nothing; What to do With Measurements Below the Detection Limits" (Ref. 4 and Ref. 6)

5.0 Test Procedures

Details of the proposed test series and procedures are presented in the Test Design Plan (TDP), and in the LOIs of the supporting contractors and laboratories. A schematic diagram of the equipment and instrument array in the BB interior is shown in Figure 1. Also, Figure 2 presents a schematic diagram of the instrument array in the BB airlock. Further, Table 3 is a matrix summary of the munitions testing and analysis. (Figures 1 and 2 and Table 3 are taken from the Test Design Plan.) Of particular interest from a quality assurance standpoint are the travel blanks and the collection of the background air samples, taken before the series of three trials for each munition item.

5.1 Detonation/Burn Set-up

Arrangements of the various munition materials for initiation and detonation or burn testing within the BB will be approved by the designated on-site explosives ordnance disposal (EOD) expert. The general layout of materials, equipment, and measurement instruments are shown in Figures 1 and 2, except that the suppression and witness shields were removed for this SERDP series (Figure 1), and the continuous HCl instrument and the method 26 sampler for HCl were added (Figure 2).

Separate samplings will be accomplished for each detonation or burn except for the residue/fallout sample, which will be a composite of the residue from the three trials for each munition. The cumulative collection of particulate from the three detonations or burns may provide a sufficient quantity of particulate mass for extraction and thus enhance detection of trace organics.

No soil sampling is planned.

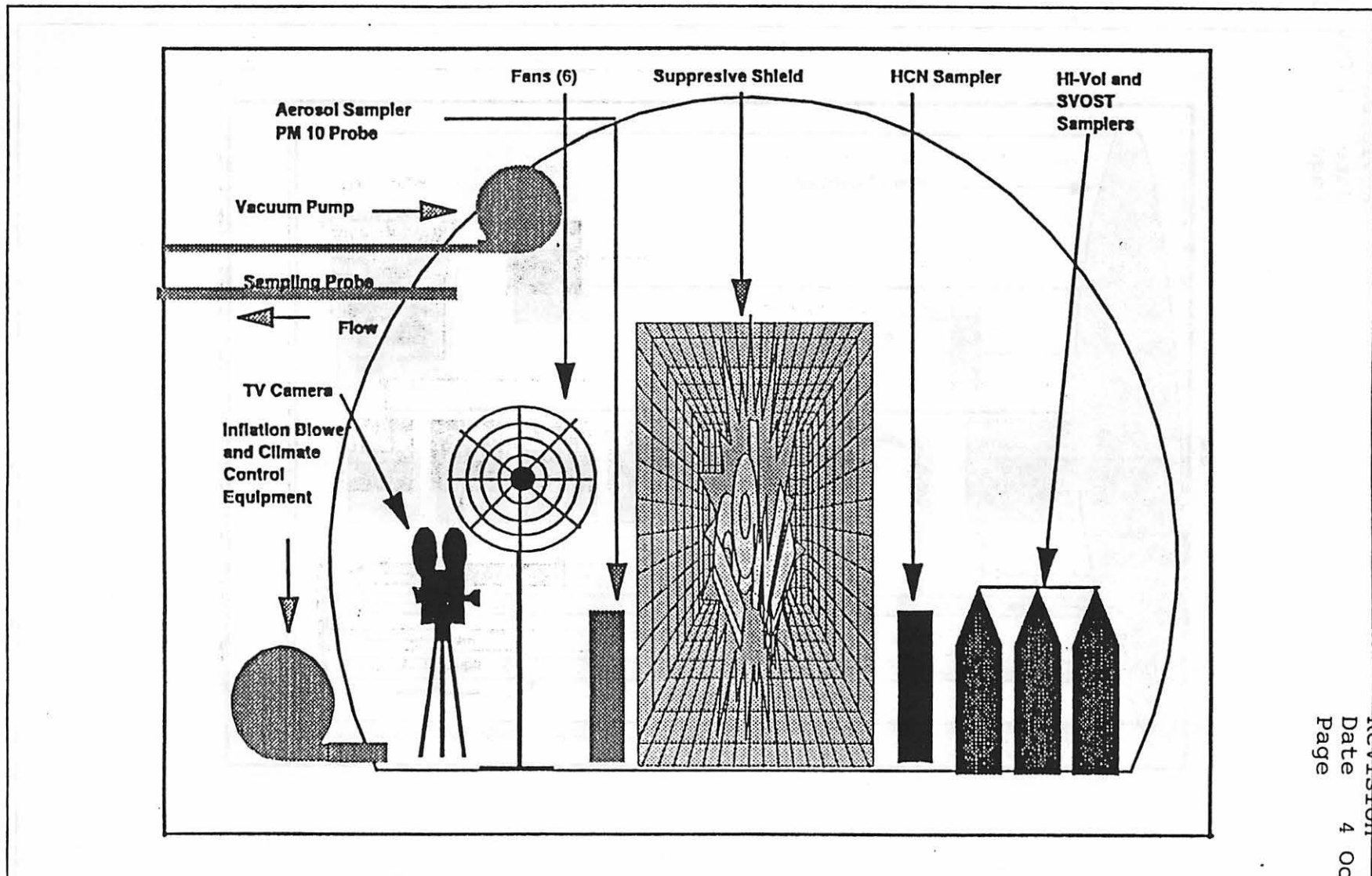


Figure 1. Schematic Diagram of Equipment and Instrument Array in the BB Interior.

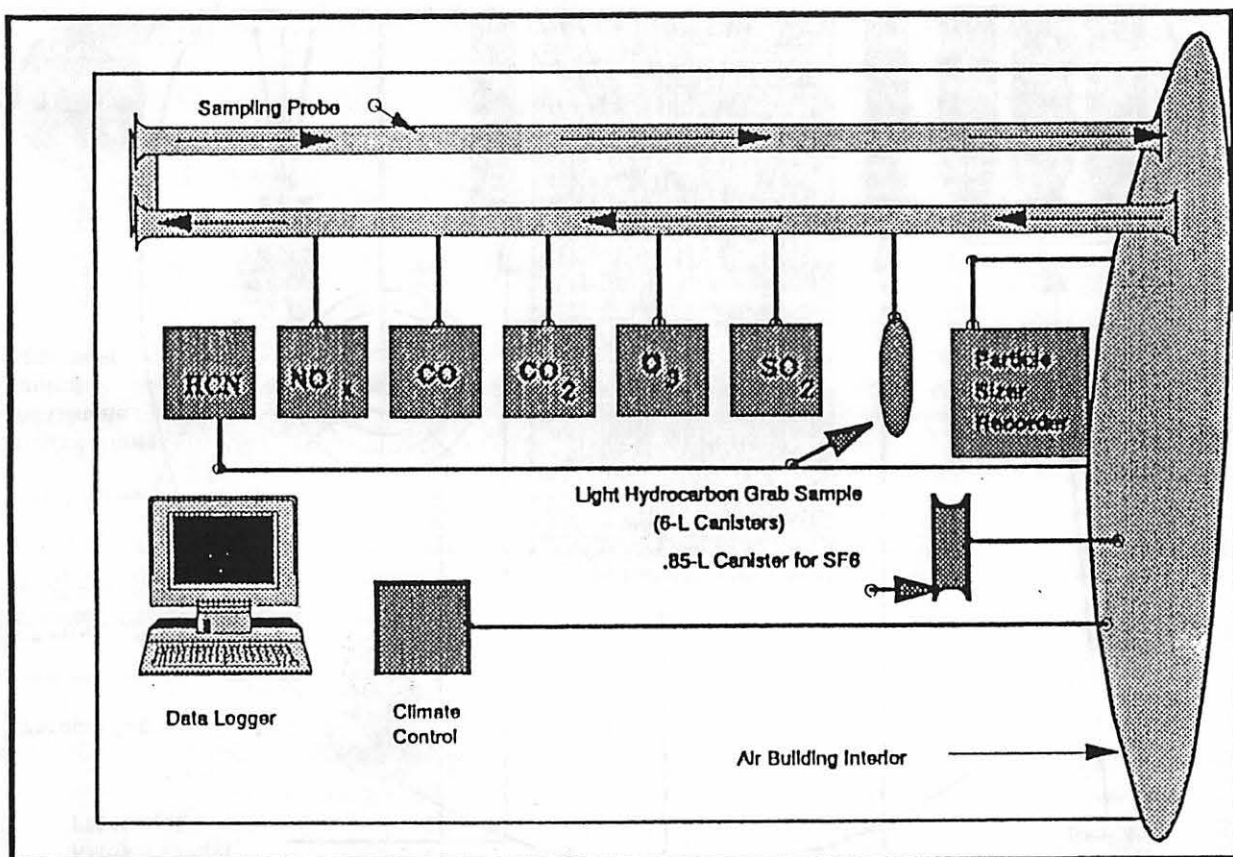


Figure 2. Schematic Diagram of Instrument Array in the BB Airlock.

Table 3 Energetic Material Testing Sampling/Analytical Matrix

Sample Source	Type of Analysis	Collection Media	Number of Samples in BB			Number of Samples Outside of BB	Number of Assays
			Travel Blank	Background	Test		
TSP High-volume air sampler	SFC/MS	Quartz filter	3	10	30	0	43
	GC/MS		3	10	30	0	43
	Metals and Carbon		3	10	30	0	43
PM-10 High-volume air sampler	GC/MS, metals	Quartz fiber filter	0	10	30	0	40
6-L Canister	Volatile organics (TO-12 and TO-14), CH ₄ , CO ₂ , and CO	SUMMA™ canister	0	30	30	10	70
PS-1	SVOC	Quartz filter; XAD-2™	3	10	30	0	43
PS-1	Dioxins	Quartz filter; XAD-2™	1	3	3	0	7
0.85-L Canister	SF ₆ , CO ₂ , and CO	SUMMA™ canister	0	30	150	0	180
Real-time analyzers	NO _x , CO, CO ₂ , O ₃ , SO ₂ , HCL			10	30	0	40 (Data logger files)
Impregnated strips	HCN or Cl ₂	Impregnated strips		10	30	0	40
Burn pan residues (for burns only)	CHN, metals, weight, SFC/MS, GC/MS	Burn pan			10*		10
Parent material	CHN						
Munitions residues (pan bottoms)	TCLP Metals & Organics Analysis (Method 8270)			0	10	0	10

*Composite sample: (1 sample/detonation trial) × (3 detonation trials/test) = 3 samples/test = 1 composite sample

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5.2 Sampling/Analysis

A combination of sampling techniques will be used to determine gaseous emissions and particulate generated from each test. These procedures will collect samples for subsequent laboratory assay and also provide real-time/near-real-time analysis. Sampling devices will be sufficiently rugged to withstand the rigors of sampling and, to the maximal extent possible, be EPA-certified. Particulate detectors and samplers are described in detail in the TDP and the LOIs.

Details of the test procedure for the BB are centered around instrument set-up, calibration, background, and air sampling. Parameters to be measured by real-time instruments include CO₂, CO, NO_x, O₃, SO₂ and particulate size distribution (PM-10). For the trials involving the burning or detonation of chlorinated materials, a real-time HCl analyzer will be used, and for comparison purposes, an EPA Method 26 impinger sampler will be employed.

Filters will be used to collect particulate and to trap semi-volatile compounds, and metals. Volatile compounds will be sampled from the BB using 6-liter canisters.

The following analytical methods will be used to analyze for metals

of interest on filters used for the collection of particulate:

EPA-SW-846-6010, Inductively coupled plasma atomic emission spectrometry (ICP) for aluminum, antimony, arsenic, barium, cadmium, chromium, copper, lead, nickel, potassium, sodium, and zinc.

EPA-SW-846-7471, cold vapor atomic absorption spectrometry (CVAA) for mercury.

EPA-SW-846-7000, flame atomic absorption spectrometry (FAA) for titanium.

Chemical analysis will be conducted by qualified laboratories as required. Procedures used will correspond with those delineated in LOIs prepared for the ACC Series II study and included in the Appendices of the TDP. The laboratories will make every effort to process samples expeditiously. Appropriate procedures for preservation of samples and materials in progress shall be implemented. Participating laboratories will be audited by QA/QC personnel.

Analyses for volatile and semi-volatile organics will be conducted by post test analytical procedures. A combination of instruments will be used to conduct the analyses at separate laboratory facilities. Instruments to be used include: Gas Chromatography (GC), Gas Chromatography/Mass Spectrometry (GC/MS), and Supercritical Fluid Chromatography/Mass Spectrometry (SFC/MS),

Samples of any residue/fallout remaining in the BB after completion of the three trials of each munition will be collected and analyzed if sufficient material is obtained. The details of the residue/fallout sample collection, sample handling, sample preservation, and laboratory analyses are included in the ECO LOIs. In addition, samples of the main explosive ingredients of some of the munition items may be analyzed.

The sweepings of munitions residues and fallout for the daily post-test cleaning will be properly labeled and archived in a freezer at DPG or the Weapons Storage Area (WSA). The sweepings will be accumulated throughout the entire series of individual detonations, for possible analysis if the total quantity is sufficient.

6.0 Test Samples

6.1 Sample Custody

Samples collected during the SERDP will be assigned unique and unambiguous serial numbers based upon the type of sample. These unique serial numbers will be assigned to the samples at the BB by designated responsible individuals from the participating laboratories. Each laboratory will be responsible for original and any internal numbering scheme used for the samples entering their laboratory. Positive assurance will be provided of the valid correlation between the specific origin of each sample and its analysis. In the event of sample splitting or compositing, the

laboratory performing these operations shall appropriately re-label and identify such samples, maintaining traceability of the materials.

6.2 Storage of Unused Samples and Unused Sample Extracts

All filters will be sealed in glass containers as soon as possible after each test and delivered to the appropriate laboratory. Glass bottles containing 8" x 10.5" filter samples are stored with dry ice prior to delivery to the laboratories. In the laboratories, prior to and after extraction and analyses, filter samples and extracts will be properly labeled and properly stored in a refrigerator or freezer. All of the above-mentioned materials will be kept properly stored in refrigerators (at $4^{\circ}\pm 2^{\circ}$) or freezers (at $-15^{\circ}\pm 5^{\circ}$) until the program is completed, i.e., until the final report has been issued, or earlier if so directed by the Test Director.

7.0 Calibration Procedures

All continuous monitors for the appropriate gases will be calibrated according to the general manufacturers' recommended procedures, and any additional requirements described in the TDP or equipment-specific LOIs. Calibration curves for selected organic exotics and inorganics will also be prepared. Quality criteria for

the appropriate calibration curves are outlined by each laboratory and recorded as parts of their LOIs. Depending on the laboratory and where appropriate, correlation coefficients and/or tables of residuals may accompany each set of data. The frequency of calibrations are noted as parts of the LOIs. In addition, technical instruments such as balances and chromatographs are calibrated periodically within specified time periods documented in LOIs. The quality criteria for calibration and maintenance of the equipment will be maintained by the individual laboratory's QC program. Daily logbooks will be kept on all instrumentation.

8.0 Data Management and Archiving of Raw Data

The accuracy of acquisition, reduction, transmittal, evaluation, and archival of all raw data obtained for test and quality assurance purposes from all field and laboratory operations is critical to the success of the SERDP study.

8.1 Test Data

The original of all technical documents and data will be sent by the originators to ECO, L. C., Attention: Cecil Eckard. Necessary copies will be made by ECO for official distribution to need-to-know individuals/organizations. ECO will maintain the official archived file until completion of the SERDP test program.

Following completion of the program, at the discretion of the Test Director, the complete archived file will then be transported to DPG for final and official storage.

The above-mentioned documents and data include all of the data collected during the field and laboratory operations, including all calibration data and logbooks. The originator of all raw data files, photographic and meteorological records, or other working records relating to technical data of the program, if not sent to ECO, will maintain such records in a secure file for at least 3 years after completion of the program.

At the completion of the final reporting of the SERDP study, ECO will prepare a list of the various types of technical data and documents, and indicate for each (a) the specific physical location of the file and (b) the custodian of the file.

8.2 Quality Assurance/Quality Control Data

To evaluate the technical data from a quality assurance standpoint for the final report, ECO or the QA consultant may request any additional quality assurance/quality control data and quality audit information from the originators, if not previously sent to ECO.

9.0 Data Analysis, Validation, and Reporting

Technical data analysis will be the responsibility of ECO. The data produced at each laboratory will be received by ECO. Once the final report is completed, ECO personnel will spot check the raw data against that which is found in the final report. All Quality Assurance/Quality Control data and reports will be evaluated by the ECO QA Consultant and will be included in the Quality Assurance Chapter of the Final Report for the SERDP study.

10.0 Internal Quality Control Checks, Audit Samples, and Frequency

The laboratory LOIs include the details of their internal quality control systems. Some of the pertinent quality control provisions of the LOIs are presented in the following sections.

10.1 BangBox Background and Volume Determination.

BangBox Background. To measure the air in the BB prior to each munition test series, all samplers and real-time instruments will be operated for 35 minutes. These results will be used as a baseline for the results obtained for each test series.

Volume Determination (Analysis of SF₆). The volume of the BangBox is determined by releasing a known amount of tracer gas,

SF₆, at the time of detonation and then determining the concentrations during the 35-minute sampling period when the gases in the BangBox are thoroughly mixed. The analysis of SF₆ is described in MSAI LOIs 022 and in OGI's SOP. However, the OGI laboratory will perform the analyses in accordance with the specifications on Table 9 of their SOP. Standards will be run until a 1% precision is achieved. During analysis periods, re-standardization is done every 15 minutes.

10.2 Real-time Measurements

Real-time gases (CO, CO₂, O₂, NO_x, SO₂, and PM-10. All real-time gas analyses will be conducted on equipment mounted within the BB. DPG LOIs 2 contains extensive detail on gas and aerosol instrument calibration conducted by DPG personnel. Real-time gas and particulate analysis is under the direction of Robert Allen of ECO, L.C.

Daily zero and single-point span checks on the instruments performance will be carried out using certified gases and by completing multi-point calibration curves at least once, prior to the entire test series and anytime the daily span exceeds $\pm 10\%$. All of the accuracy data must fall within $\pm 10\%$ of the known amount, or corrective action must be completed. Following any corrective action, another multipoint calibration is performed. At

the end of the entire series of tests, post-test zero and span checks are performed.

Precision estimates (relative standard deviations) from the same day should fall within 15%.

Analysis of HCN, HCl, Cl₂. The analyses for HCN, HCl, and Cl₂ are described in DPG LOI 2, using a Continuous Toxic Gas Monitor utilizing a continuous chemically-impregnated paper strip prepared by the manufacturer of the instrument. The paper strip increases in depth of color with increasing concentration. At the BangBox site, the instrument will be optically calibrated using a color card according to the manufacturer's instructions. The instrument will be used to monitor HCN for all detonations except for the munitions containing chlorine, when either HCl or Cl₂ will be monitored. Before each trial, a prior decision must be made for which one of the three chemicals is to be monitored. For the SERDP series of trials involving chlorinated materials, HCl will be also be measured by (1) a Gas Filter Correlation analyzer, and (2) EPA Method 26 impinger sampler.

10.3 Laboratory Analyses

Each laboratory has an internal QC program. These QC programs will be documented along with a detailed description of the operations

in the LOIs. Basically, the QC checks are a listing of the operational checks, the control limits for initiating corrective action, and the planned corrective action. Examples of items that are included:

- a. Replicates
- b. Spiked samples
- c. Split samples
- d. Control charts
- e. Blanks
- f. Internal standards
- g. Quality control samples
- h. Calibration standards and devices
- i. Reagent checks

Of particular importance is the use of control charts, the plotting of quality control data in chronological order, with appropriate statistical control limits. Such charts should be maintained on each appropriate analytical process or unit of equipment for the entire project. These charts show quantitatively the degree of variation of the process or instrument through time, and they provide a permanent record of this variation. Table 4 lists the QC/audit checks and the criteria for acceptable performance limits.

Details of these internal QC programs are provided in the laboratories' LOIs (Appendices D through J). The sections below present summaries of the QC/audit procedures for each task in the SERDP study.

Table 4. Quality Control, audit checks, and criteria for the SERDP study.

Measurement	Type of Procedure	Frequency	Criteria
Hi-vol filter weights (before and after sampling)	weighing filters	100%	$\pm 3s$
GC/MS semivolatiles (filters)	filter blanks trip blanks calibration blanks filter spikes (LCS) filter spike duplicates (LCSD) calibration checks	1/extraction event 1/sampling week 10% 1/20 1/20 1/12-hr period	< quantitation limit < quantitation limit < quantitation limit recovery limits: 8270A, Table 6 precision limits: 8270A, Table 6 CCC response factor % Diff < 30% SPCC response factor > 0.05
SFC/MS semivolatiles (filters)	filter blanks trip blanks calibration blanks filter spikes (LCS) filter spike duplicates (LCSD) calibration checks	1/extraction event 1/sampling week 1/day 1/20 1/20 10%	< quantitation limit < quantitation limit < quantitation limit $\pm 50\%$ of known concentration absolute value < 50% RPD. response factor % Diff < 50%
metals (filters)	method blanks trip blanks calibration blanks calibration checks matrix spikes analysis duplicates laboratory control samples	1/20 1/sampling week 10% 10% 1/20 1/20 1/20	< method detection limit < method detection limit $\pm 3s$ $\pm 10\%$ of known concentration $\pm 20\%$ of known concentration absolute value < 20% RPD $\pm 20\%$ of known concentration
volatiles (6 Liter canisters)	GC-FID calibration with Neohexane daily NIST VOC standard duplicate samples EPA spike	once once 10% several	$\pm 2\%$ of known concentration 15-20% of known concentration $\pm 2\%$ of known concentration $\pm 5\%$ of known concentration
real-time gases	calibration zero/span checks performance audit	prior to and after tests daily once	$r^2 > 0.98$ $\pm 10\%$ $\pm 15\%$ of known concentration
toxic gas monitor	calibration (optical) zero/span checks (electronic)	prior to and after tests and after any change in cassettes daily and when changing cassettes	K-factor within specified range Q-factor within specified range
real-time PM-10	calibration (according to manufacturer's manual)	prior to and after tests	$\pm 2\%$ of specified level
Method 26 impinger (HCl)	calibration (flow rate) calibration (chemical)	prior to each trial prior to each lab batch	???? ????

10.3.1 Hi-Vol Quartz Filters

Filter Handling, Weighing and Storage. The QC program for the handling, weighing, and storage of filters is included in MSAIs LOIs 002.

Semivolatile Organics (filters). Analysis of the semivolatile organics from particulates will be under the direction of Dr. Doug Later of MSAI. MSAI's QC program is described in their LOIs.

Filter Blanks. Travel blanks are to be used to determine the effects, if any, of the movement of the filters from the laboratory to the BB, the holding of the filters at the BB during the testing in closed containers (the same type of container used to transport the exposed filters), and the return to the laboratory. Analysis of the travel blank results will reveal the extent, if any, of any pick-up (adsorption/absorption) of organic materials. Results of the travel blanks will be compared with the results of filter blanks that "do not travel", i.e., those that remain in the laboratory.

Extraction Completeness. To evaluate extraction completeness, one real sample from two different munition items will be extracted twice with the specified extractant. Based on the results, additional extractions of other samples may or may not be necessary. (Acetonitrile has been demonstrated in the literature {Jenkins and Grant, Ref. 3} to be the preferred solvent.)

Instrument Calibration and Standardization. Instrument Quality control for the SFC/MS and GC/MS system will be conducted in several different ways.

Calibration plots for the target analytes will be prepared at the start of the study. From these plots, the detection limits and quantitation limits are calculated for each target analyte. Detection limits for the metals and GC/MS target analytes are determined by the method given in 40 CFR 136, Appendix B. SFC detection limits are determined by dilution of standards to the concentration at which the standard is detected at or slightly greater than 3 times the background noise. Quantitation limits are determined as multiples of the detection limits. The multiplier used for each analyte or class of analytes is selected to account for factors present in normal analysis conditions. These quantitation limits are presented in Tables 4A and 4B.

Table 4A. Quantitation Limits for Target Analytes determined by Chemical Ionization (CI)/Selected Ion Monitoring (SIM)/SFC/MS and SFC/Chemiluminescence detection (TEA)

Compound	Quantitation Limit (ng μL^{-1})
Nitroglycerine	1
2,6-Dinitrotoluene	1
N-Nitrosodiphenylamine	1
2,4-Dinitrotoluene	1
Pentaerythritol tetranitrate (PETN)	1
2-Nitronaphthalene	1
2,4,6-Trinitrotoluene	1
2-Nitrodiphenylamine	1
1,3,5-Trinitrobenzene	1
N-Methyl-N,2,4,6-tetranitroaniline (TETRYL)	1
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	1
1-Nitropyrene	1
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	1
Naphthalene	1
Phenol	1
Dibenzofuran	1
Diphenylamine	1
Pyrene	1
Benz[a]anthracene	1
Benzo[a]pyrene	1

Table 4B. Quantitation Limits for Target Analytes by GC/MS under Electron Ionization

Compound	Quantitation Limit (ng μL^{-1})
Phenol	1
bis(2-Chloroethyl) ether	1
2-Chlorophenol	1
1,3-Dichlorobenzene	1
1,4-Dichlorobenzene	1
Benzyl alcohol	2
1,2-Dichlorobenzene	1
2-Methylphenol (o-Cresol)	1
bis(2-chloro-1-isopropyl) ether	1
3 + 4-Methylphenol (m+p cresol)	1
Pentachloroethane	1
N-Nitrosodi-N-propylamine ¹	1
Hexachloroethane	1
Pyridine	1
N-Nitrosodimethylamine ¹	1
2-Picoline	1
N-Nitrosomethylethylamine ¹	1
Methyl methanesulfonate	1
N-Nitrosodiethylamine ¹	1
Ethyl methanesulfonate	1
Aniline	1
N-Nitrosopyrrolidine ¹	1
Acetophenone	1
N-Nitrosomorpholine ¹	2
o-Toluidine	1
Nitrobenzene	1

Isophorone	1
2,4-Dimethylphenol	1
2-Nitrophenol	1
bis(2-Chloroethoxy)methane	1
2,4-Dichlorophenol	1
1,2,4-Trichlorobenzene	1
Naphthalene	1
4-Chloroaniline	2
Hexachloro-1,3-butadiene	1
4-Chloro-3-methylphenol	1
2-Methylnaphthalene	1
N-Nitrosopiperidine ¹	1
o,o,o-Triethylphosphorothioate	1
2,4-Dichlorophenol	1
Hexachloropropene	1
a,a-Dimethylphenethylamine	2
2,6-Dichlorophenol	2
1,4-Phenylenediamine	10
N-Nitrosodi-N-butylamine ¹	1
Hexachlorocyclopentadiene	5
2,4,6-Trichlorophenol	1
2,4,5-Trichlorophenol	1
2-Chloronaphthalene	1
2-Nitroaniline	5
Dimethyl phthalate	1
2,6-Dinitrotoluene	1
Acenaphthylene	1
3-Nitroaniline	5
2,4-Dinitrophenol	5
Acenaphthene	1
4-Nitrophenol	5

2,4-Dinitrotoluene	1
Dibenzofuran	1
Diethyl phthalate	1
4-Chlorophenyl-phenyl ether	1
Fluorene	1
4-Nitroaniline	2
1,2,4,5-Tetrachlorobenzene	1
Isosafrole	1
Safrole	1
1,4-Naphthoquinone	5
1,3-Dinitrobenzene	1
Pentachlorobenzene	1
1-Naphthylamine	1
2,3,4,6-Tetrachlorophenol	1
2-Naphthylamine	2
Thionazin	2
5-Nitro-o-toluidine	1
4,6-Dinitro-2-methylphenol	5
N-Nitrosodiphenylamine ¹	1
4-Bromophenyl-phenyl ether	1
Hexachlorobenzene	1
Pentachlorophenol	5
Phenanthrene	1
Anthracene	1
Di-N-butyl phthalate	1
Fluoranthene	1
Tetraethyl dithiopyrophosphate	1
1,3,5-Trinitrobenzene	5
cis-Diallate	1
trans-Diallate	1
Dimethoate	5

Phenacetin	1
4-Aminobiphenyl	1
Pentachloronitrobenzene	1
Pronamide	1
4-Nitroquinoline-1-oxide	5
Methapyrilene	5
Isodrin	1
Pyrene	1
Butylbenzyl phthalate	1
bis(2-Ethylhexyl) phthalate	1
3,3'-Dichlorobenzidine	2
Benz(a)anthracene	1
Chrysene	1
p-(Dimethylamino)azobenzene	1
Chlorobenzilate	1
3,3'-Dimethylbenzidine	1
2-Acetylaminofluorene	1
Di-N-octyl phthalate	1
Benzo(b)fluoranthene	1
Benzo(k)fluoranthene	1
Benzo(a)pyrene	1
Indeno(1,2,3-cd)pyrene	1
Dibenz(a,h)anthracene	1
Benzo(ghi)perylene	1
7,12-Dimethylbenz(a)anthracene	1
Hexachlorophene	1
3-Methylcholanthrene	1
Perylene	1
Biphenyl	1
N-Nitrosodimethylamine ¹	1

1. N-Nitroso compounds are degraded by the hot gas chromatograph injector. In each case, the new molecule, formed by replacement of the N-nitroso functional group with hydrogen, is chromatographically separated and mass spectrometrically detected. For example, N-nitrosodiphenylamine degrades to diphenylamine.
-

Instrument calibration will be conducted daily prior to analyzing samples.

Standard solutions of analytes will be run daily and compared with the calibration plots to ensure adherence to the calibration plot. These values will be plotted on control charts chronologically. If the values deviate or drift significantly from the calibration value $\pm 3s$, then the analyte solution and calibration will be redone. Analytical Precision. Precision from the test filter samples will be estimated. Repeated aliquots from the same filter extract will be injected into the SFC/MS (and a few GC/MS) system to get an estimate of the analytical error (imprecision). These values should be within 10% of each other.

Duplicate and Spiked Digestates for Metals Analyses. Periodically -- at least once in every 20 samples -- two sample filters will be designated for quality control, one for duplicate analysis and the other for spike recovery analysis. From each individual filter, two duplicate sections will be digested and analyzed separately. The sample designated as the spike will be

evaluated for the percent recoveries of spiked analytes added to one of the duplicate sections. The sample designated for duplicate analysis will be used to evaluate analytical precision by determining relative percent difference (RPD) between results of the duplicate sections. The recovery and RPD results will be technically reviewed against acceptance limits and appropriate corrective actions will be taken when the limits are exceeded.

Laboratory Control Sample (LCS) and LCS Duplicate (LCSD) for Organics Analyses. Periodically -- at least once with every 20 samples -- two pairs of clean "blank" filters will be spiked with known amounts of each target compound. Each pair of filters (designated LCS and LCSD) will be extracted and analyzed separately. The sample designated as the LCS will be evaluated for the percent recoveries of spiked compounds. The relative percent difference (RPD) between results of the LCS and LCSD will be used to evaluate analytical precision. The recovery and RPD results will be technically reviewed against acceptance limits and appropriate corrective actions will be taken when the limits are exceeded.

Metals. Metals of interest in particulate filters will be analyzed by inductively coupled plasma atomic emission spectrometry (ICP) for aluminum, antimony, arsenic, barium, cadmium, chromium, copper, lead, nickel, potassium, sodium, and zinc; cold vapor atomic absorption spectrometry (CVAA) for mercury; and flame atomic absorption spectrometry (FAA) for titanium. Accuracy will be

determined by analysis of NIST SRM's. Results from the SRM's should be recoveries that are within $\pm 10\%$ of the assessed SRM value. If the data do not come within these limits, then the measurement system will be evaluated and appropriate corrective action will be taken.

Precision will be determined by analyses of duplicate filter sample sections. Quantitation limits for the ICAP[®] analysis vary from 1.5 to 250 $\mu\text{g}/\text{filter}$ (5 to 750 $\mu\text{g}/\text{L}$). For the primary elements of interest the quantitation limits are:

Al	40 $\mu\text{g}/\text{filter}$ (125 $\mu\text{g}/\text{L}$)
Sb	110 $\mu\text{g}/\text{filter}$ (350 $\mu\text{g}/\text{L}$)
As	35 $\mu\text{g}/\text{filter}$ (100 $\mu\text{g}/\text{L}$)
Ba	2 $\mu\text{g}/\text{filter}$ (5 $\mu\text{g}/\text{L}$)
Cd	5 $\mu\text{g}/\text{filter}$ (15 $\mu\text{g}/\text{L}$)
Ca	160 $\mu\text{g}/\text{filter}$ (500 $\mu\text{g}/\text{L}$)
Cr	25 $\mu\text{g}/\text{filter}$ (75 $\mu\text{g}/\text{L}$)
Cu	8 $\mu\text{g}/\text{filter}$ (25 $\mu\text{g}/\text{L}$)
Pb	50 $\mu\text{g}/\text{filter}$ (160 $\mu\text{g}/\text{L}$)
Hg	0.06 $\mu\text{g}/\text{filter}$ (0.5 $\mu\text{g}/\text{L}$)
Ni	25 $\mu\text{g}/\text{filter}$ (75 $\mu\text{g}/\text{L}$)
K	200 $\mu\text{g}/\text{filter}$ (500 $\mu\text{g}/\text{L}$)
Na	250 $\mu\text{g}/\text{filter}$ (750 $\mu\text{g}/\text{L}$)
Ti	1.5 $\mu\text{g}/\text{filter}$ (5 $\mu\text{g}/\text{L}$)
Zn	35 $\mu\text{g}/\text{filter}$ (100 $\mu\text{g}/\text{L}$)

ICAP[®] is a trademark of Thermo Jarrell Ash Corporation.

10.3.2 Semi-volatile organics (XAD-2 Cartridges).

Analysis of the semi-volatile organics will also be performed using the PS-1 sampler, which contains an XAD-2 resin cartridge. The detailed procedures are specified in Radian LOIs 26 and 27.

Travel Blanks. Travel blanks for the XAD-2 cartridges will be used in a similar manner as that described above for travel blank filters.

Sampler Flow Rate Calibration. Prior to each munition test series the sampler flow rate will be calibrated using a dry gas meter. The r^2 for each calibration should exceed 0.9998.

Spiked Extracts. Accuracy for extraction of analytes from the XAD-2 cartridges will be assessed in the following manner. An unexposed cartridge will be spiked with a high concentration of analytes and one with a "lower" concentration of analytes. The high and lower concentrations will be selected to bracket the expected levels found in real test samples. All percent recovery results should be within $\pm 50\%$ of the amount added. If these values fall outside these limits, then an appropriate corrective action will be needed. These checks should be performed once during the early period of the analyses and once during the latter part of the period.

Duplicate Extract Analyses. Once for the samples for each munition test series, the extract should be split, with the analyses for the splits performed on different days.

10.3.3. Volatiles (6-L Canisters). Volatile organics are trapped in stainless steel 6-L canisters located in the BB. Analysis of these volatile organics is under the direction of R.A. Rasmussen of OGI. A QC program is outlined in OGI's Standard Operating Procedure (LOIs).

Travel Blank Canisters. To provide a baseline against which the real samples may be compared, travel blank canisters are transported to and from the testing site along with the sample canisters and are scheduled through the analysis at the OGI laboratory.

Calibrations. The analytical systems used to analyze the SERDP samples are calibrated as needed. For metals analysis, calibration is performed before every analysis run and again when calibration checks fail to meet acceptance criteria. GC/MS, SFC/MS, and SFC/TEA are calibrated before the initial analysis and again when calibration checks fail to meet acceptance criteria. Acceptance criteria for calibration checks are summarized in Table 4.

Analytical System Blanks. Analytical system blanks will be used periodically to assure that the instrumental system has not become contaminated.

Recovery Efficiency. Duplicate sections of specific samples for metals analyses will be digested separately, with one digestate being spiked to determine how well the spike is recovered. For organics analyses, a laboratory control sample (LCS), which is a filter blank spiked with the compounds of interest, will be analyzed to determine spike recovery. Percent recovery is used to measure analytical system efficiency.

Precision Checks. Precision is evaluated by analyses of split samples or by analyses of an LCS and an LCSD. A precision check is performed at least once for every 20 samples. Duplicate precision is measured in terms of relative percent difference (RPD). The results for metals must be within ± 20 RPD. Duplicate acceptance criteria for organics analyzed by SFC/MS are ± 50 RPD. The criteria for semivolatile organics by GC/MS vary by compound according to statistically derived control limits.

Detection Limits. Detection limits for C₁ to C₁₀ compounds will be 0.1-0.2 ug/m³ depending on the compound.

Performance and System Audits. Accuracy of the analytical system will be assessed by analyzing VOC standards from NIST and audit cylinder from EPA. Percent recovery results should come within 15-20% of the amount added, or corrective action of repeat

samples may be recommended. Further, personnel from the EPA/RMEB/RTP will visit the OGI laboratory to conduct a systems audit of the laboratory's operations.

OGI Reported Results. The following excerpts from the OGI LOIs present the demonstrated quality of their analytical systems.

TABLE 5. OGI-REPORTED DATA QUALITY

<u>METHOD</u>	<u>SPECIES</u>	<u>DETECTION LIMITS</u>	<u>PRECISION</u>
Carle 211-M-FID	CH ₄	5 ppb	@ 1700 ppbv $\pm$ 0.2%
	CO	10 ppb	@ 100 ppbv $\pm$ 3.0%
	CO ₂	0.05 ppm	@ 345 ppbv $\pm$ 0.3%
Perkin Elmer 3920/ECD	Air toxics	< 10 pptv	@ 100 ppbv $\pm$ 1%
	Halocarbons	< 1 pptv	@ 10 ppbv $\pm$ 5%
	N ₂ O	< 10 ppbv	@ 300 ppbv $\pm$ 1%
Hewlett-Packard 5790/FID	C ₁ -C ₈	10 pptv	@ 100 pptv $\pm$ 10%
	Hydrocarbons	1000 mL	@ 1000 ppbv $\pm$ 2.0%
Hewlett-Packard 5890/FID	TNMHC	< 0.1 ppbv	@ 1 ppbv $\pm$ 10%
	C ₇ -C ₁₀	< 0.1 ppbv	@ 1 ppbv $\pm$ 10%

11.0 Performance and System Audits

Personnel from ECO L.C. (QA Consultant) and from EPA/RTP, RMEB, will visit MSAI and other labs while SERDP analyses are being performed to monitor and assess the capability and performance of all measurement and analysis systems, and assess the adherence to approved procedures. The Laboratory Director will be contacted prior to the visit. Appropriate reports will be sent to the ECO Project Manager. A copy of the audit review reports will be

retained in the ECO project files.

Normally, an EPA Audit-Team would provide a Performance Audit on the real-time instruments in the BB, including NO_x, NO₂, NO, CO, CO₂, O₃, HCl, and sampler flow. Likewise, external performance audit samples would normally be prepared for the analytical laboratories -- OGI, Radian, and MSAI. However, as mentioned in Section 3, because of the very satisfactory results obtained for the ACC-2 project just recently from the same laboratories performing the same analyses, it was deemed sufficiently valid to consider the previous audits as applicable to this study.

12.0 Preventive Maintenance

Preventive maintenance tasks and schedules recommended by the manufacturers of the gas analyzers, chromatographs, mass spectrometers, and other technical equipment will be followed or exceeded. Considering the importance of the data for this project, more stringent schedules may be deemed appropriate. Spare parts such as detector heads, septa, columns, and cylinder gases should be maintained on-hand during the project for equipment repair to minimize downtime.

13.0 Specific Procedures to be used to Routinely Assess Data Quality

Specific procedures, to be detailed in the LOIs, will be used to assess data quality.

13.1 Specific Procedures to be used to Routinely Assess Data Precision and Accuracy

There are routine procedures used to assess the precision and accuracy of the measurement data. Each analytical laboratory will use procedures appropriate for its analytical measurement systems. The detailed operational and computational procedures should be specified in its respective LOIs. Estimates of precision that involve or include sampling variability as well as analytical variability, such as the use of duplicate samples will be made by ECO L.C. during the data evaluation process. In fact, the agreement of results among the triplicate trials for each munition is a measure of the overall precision of the entire test system, including the sampling and analysis operations.

Appropriate statistical procedures for the detection of outliers will be used by ECO, L.C. during the data evaluation process. Investigation of any outliers and decision concerning the use of outlier data will be made by ECO in conjunction with appropriate members of the Technical Steering Committee.

Procedures for determining detection limits will be specified by the laboratories in their LOIs.

13.2 Specific Procedures to be Used to Routinely Assess Data Completeness

Completeness of the data acquired during the test series will be computed from two standpoints. One is the completeness with respect to sampling. The second is the completeness with respect to analysis. Completeness with respect to sampling is the number of valid samples -- obtained at the site in the case of continuous real-time measurements, or delivered to the laboratories in the case of laboratory analyses -- compared to the total number of planned samples, expressed as a percentage. Completeness with respect to analysis is the number of valid analyses obtained from samples delivered to the laboratory compared to the total number of planned analyses, expressed as a percentage.

14.0 Quality Assurance Reports

Reports, covering the quality assurance activities of each laboratory will be provided by the laboratory managers to the ECO Project Manager. Reports will also be made of the quality assurance activities of the various field operations and will be provided by the appropriate field manager to the ECO Project Manager. Results of the blind performance audits and systems

audits conducted by the EPA/RTP/RMEB will be reported to the ECO Project Manager. These reports may include:

- Assessment of measurement data accuracy, precision, detection limits, and completeness.
- Results of performance audits
- Results of system audits
- Significant QA/QC problems and recommended solutions

ECO, L.C. will in turn forward all Quality Assurance/Quality Control reports to the ECO, L.C. Quality Consultant.

A complete summary and evaluation of all quality assurance/quality control data will be included in the Quality Assurance Chapter of the Final Report for the project.

15.0 Summary

The goal of this QA Project Plan is to provide the procedures or protocols, objectives, functional activities, and specific QA/QC activities associated with the project and which are designed to achieve acceptable data quality. The major objectives of this QA Plan are to ensure the quality of the field and laboratory data, and to document the QA/QC procedures for the overall study. An important part of the QA Project Plan is for each of the field and laboratory organizations to prepare and have available at the work sites adequate LOIs to establish the written, documented procedures used by each organization. These LOIs should establish the

calibration procedures, including the standards used for calibration, frequency of calibrations, and acceptability of calibration results. The LOIs should also include a description of all the routine QC activities.

The flow of all technical data and documents will be from each organization to the ECO Project Manager as described in Section 8.1. During the work effort, there will be site visits to field and laboratory activities to assess QA/QC compliance.

16.0 Glossary of Terms

Audit - A systematic check to determine the quality of operation of some function or activity. Audits may be of two basic types: (1) performance audits in which quantitative data are independently obtained for comparison with routinely obtained data in a measurement system, or (2) system audits of a qualitative nature that consist of an on-site review of a laboratory's QA/QC system and physical facilities for sampling, calibration, and measurement.

Chain of Custody - A procedure for preserving the integrity of a sample or of data (e.g. a written record listing the location of the sample/data at all times).

Data Quality - The totality of features and characteristics of data that bear on their ability to satisfy a given purpose. The

characteristics of major importance are mainly accuracy, precision, and completeness. These characteristics are defined as follows:

1. Precision - A measure of mutual agreement among individual measurements of the same property, usually under prescribed, similar conditions. Precision is best expressed in terms of the standard deviation. Various measures of precision exist, depending upon the "prescribed similar conditions." The magnitudes of precision vary at different magnitudes of the characteristic/analyte being measured. Absolute levels of precision generally increase with increased levels of the characteristic/analyte being measured.

2. Accuracy - The degree of agreement of a measurement, X , with an accepted reference or true value, T , usually expressed as the difference between the two values, $X-T$, or the difference as a percentage of the reference or true value, $100(X-T)/T$, and sometimes expressed as a ratio, X/T . Absolute measures of accuracy will also vary at different magnitudes of the "thing" being measured, and will generally increase with higher levels of the "thing" being measured.

3. Completeness - A measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct, normal conditions. Completeness can be expressed separately in terms of sampling or analysis.

Data Validation - A systematic process for reviewing a body of

data against a set of criteria to provide assurance that the data are adequate for their intended use. Data validation consists of data editing, screening, checking, auditing, verification, certification, and review.

Letter of Instruction (LOI) - A written document which outlines in general an operation, analysis or action whose mechanisms are generally prescribed and which are commonly accepted as the methods for performing certain routine or repetitive tasks. The LOIs are more general than the Standard Operating Procedures (SOPs), which usually provide detailed step-by-step bench-level instructions.

Quality Assurance (QA) - The total integrated program for assuring the accuracy and reliability of monitoring and measurement data. A system for integrating the quality planning, quality assessment, and quality improvement efforts to meet user requirements.

Quality Assurance Project Plan (QAPP) - An orderly assembly of detailed and specific procedures which delineates how data of known and acceptable quality are produced for a specific project.

Quality Control (QC) - The routine application of procedures for obtaining prescribed standards of performance in the monitoring and measurement process. Often, QC procedures are referred to as those standards of performance that are checked within a laboratory.

Quality Performance Audits - Procedures used to determine

quantitatively the accuracy of the total measurement system or component parts thereof.

Quality System Audits - Procedures used to critically review and qualitatively evaluate the capability of an organization to provide data or product of acceptable quality.

17.0 References

1. American Chemical Society, Analytical Chemistry, Guidelines for Data Acquisition and Data Evaluation in Environmental Chemistry, (1980) 52, 2242-2249.
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3. Jenkins, Thomas F. and Clarence L. Grant. 1987. Comparison of extraction techniques for munitions residues in soil. Anal. Chem. 59: 1326-1331.
4. Rhodes, R. C. 1981. Much Ado About Next to Nothing, or What to Do with Measurements Below the Detection Limits, Environmetrics '81, April 8-10, 1981, Alexandria, VA.
5. Rhodes, R. C. 1988. Quality Assurance Project Plan for the Integrated Air Cancer Project (IACP) - Study Roanoke, Virginia. EPA Contract Numbers 68-02-4550. The Quality Assurance Division Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27709.
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7. Taylor, J. K. 1987. Quality Assurance of Chemical Measurements. Lewis Publishers, Inc.
8. U.S. Army Dugway Proving Ground, August 1995, Test Design Plan, Strategic Environmental Research and Development Program, Dugway, UT.

Appendix A

Procedures for Writing Letters of Instruction (LOIs)

1. Purpose

QA/QC practices require that all routine activities having an impact on data quality be fully documented. These day-to-day routine practices should be itemized in a clear, explicit, somewhat detailed, step-by-step manner so that most individuals could follow them in a uniform and consistent manner.

2. Scope

LOIs should be prepared for all routine activities associated with the OB/OD project. Such activities may include:

- Field or Laboratory Sampling
- Field or Laboratory Analysis
- Instrument or Method Calibrations
- Preventative and Corrective Maintenance
- Internal QC Procedures
- Site Selection
- Sample Preparation and Storage
- Preparation and use of Spiked Samples
- Instrument/Equipment Selection and use
- Determination of Detection Limits and Limits of Quantification
- Sample Handling and Transportation
- Conduct Performance Audits
- Data Reduction and Analysis

Once the LOIs are written, they will be reviewed for adequacy by the QA Officer and Technical Coordinator, and kept in each Laboratory's files and made available during QA visits. LOIs may also be changed or revised during the study to conform with the actual work effort. However, these changes should be clearly documented and the old LOIs replaced with the new ones (see document control section below).

Procedures for Writing Letters of Instruction (LOIs) (continued)

3. Format

The format and outline of LOIs may vary somewhat depending on the activity. However, all LOIs should contain the following:

1. Title
2. Outline of procedures
3. References (if appropriate)
4. Document control block in upper right hand corner
5. Signature line for project officer and signature line for QA

4. Document Control

Each page of the LOIs should contain a document control block in the upper right corner containing: LOI number, date of issue, revision number, and page number similar to that used for this document.

5. Where to Send LOIs

A copy of each LOI should be kept in the originating laboratory and a signed copy should be sent to:

Cecil Eckard, Project Manager
ECO, L.C.
139 East on South Temple Street
Suite 400
Salt Lake City, UT 84111
(801) 532-5149
FAX (801) 538-0073

Appendix B

Sampling Numbering System

Samples collected during the SERDP tests will be assigned unique and unambiguous serial numbers based upon the type of sample and trial, and/or time of trial. These unique serial numbers will be assigned to the samples at the BB by designated responsible individuals. Each laboratory will be responsible for original and any internal numbering scheme used for the samples entering their laboratory. Positive assurance will be provided of the valid correlation between the specific origin of each sample and its analysis. In the event of sample splitting or compositing, the laboratory or other organization performing these operations shall appropriately re-label and identify such samples, maintaining traceability of the materials.

Appendix C

Calculation of Precision, Accuracy, and Completeness

The procedures for calculating precision and accuracy (i.e., bias) follow the EPA Quality Assurance Management Staff (QAMS) guidelines in "Chapter 5 - Calculation of Precision, Bias, and Method Detection Limit for Chemical and Physical Measurements". The definition of completeness used herein is taken from the QAMS "Interim Guidelines for Preparing Quality Assurance Project Plans".

A. Precision

Precision estimates for a measurement system and/or component of the measurement system will be obtained by use of collocated (duplicate) samples or repeated measurements of reference material. Procedures for both methods are described in the next two sections.

Duplicate samples

This calculating procedure applies to the use of collocated samples for assessing the precision of the total measurement system and to the use of replicated samples for assessing precision of a component of the measurement system.

For the i^{th} sample pair X_i , Y_i (either collocated or replicated), calculate the standard deviation by

$$s_i = (X_i - Y_i)/1.414,$$

or the percent relative standard deviation by

$$\%RSD_i = 200 s_i / (X_i + Y_i).$$

A table is prepared with the results of the n sample pairs containing values of X_i , Y_i , $(X_i + Y_i)/2$, s_i , and $\%RSD$. Other means of reporting precision estimates include: $\%RSD$ values graphed versus the average values of the pairs, or a regression equation of $\%RSD$ versus the average measured values. The precision estimate or tabulated values should be identified with the applicable portion or portions of the measurement system (e.g., the total system for collocated samples or sample preparation and analysis for laboratory replicates).

Repeated Measurements of the Same Material

This calculation procedure applies to situations where the precision estimate is based on repeated measurements of a reference material (or of the same material) as is the case for continuous air quality monitors.

For n repeated measurements of the same material (e.g., the same pressurized cylinder gas), the standard deviation (s) of the responses is given by:

$$s = \left\{ \frac{1}{(n-1)} \sum_{i=1}^n (x_i - \bar{x})^2 \right\}^{1/2}$$

where

x_i = the response of the i^{th} measurement
 $\bar{x}$ = the average of the n measurements
n = the total number of measurements

and the %RSD is calculated by

$$\%RSD = (100)s/\bar{x}.$$

These calculations are performed for each level (e.g., concentration level) tested. Precision estimates may be reported as s and/or %RSD for specific concentration levels or as a function of concentration over the applicable concentration range.

B. Accuracy (Bias)

The accuracy (bias) of a measurement system or portion thereof is estimated of spiked samples or by repeated analyses of a reference material.

C. Spiked Samples

Percent recovery (P) for the i^{th} spiked sample is calculated by:

$$P_i = 100(A_i - B_i)/T_i,$$

where:

A_i = the analytical result from the spiked sample,
 B_i = the analytical results from a separate analysis of the unspiked sample,
 T_i = the known true value of the spike.

The relationship between percent bias (%B) and the average percent recovery (P) is:

$$\%B = P - 100.$$

where:

$$P = \frac{\sum_{i=1}^n P_i}{n}.$$

D. Analyses of Reference Material

Bias B is the difference between the average value X of a set of n measurements of a standard (reference material) and the accepted (or true) T value of the standard and is given by

$$B = X - T$$

and the percent bias %B is given by

$$\%B = 100(X - T)/T.$$

Bias estimates can be reported for the specific levels tested or presented graphically as a function of level or represented by a regression equation over the applicable range of levels tested.

E. Completeness

Basically, completeness for each sample collection and analysis scheme is the ratio of the number of valid analysis results to the number of samples intended to be collected and analyzed.

Sampling Completeness.

Real-time Data. In the case of real-time data, sampling completeness for each type of real-time data is (a) the number of valid data points of that type recorded and converted to engineering units compared to (b) the total number of such data points planned to be recorded and converted to engineering units.

Samples Taken for Laboratory Analysis. The sampling completeness for each type of laboratory sample will be (a) the number of valid samples of that type actually taken and delivered to the appropriate laboratory compared to (b) the total number of each such type of samples planned to be taken and delivered to the laboratory. Special allowance will be made for samples (such as intentional spares or extras) taken with a later decision being made concerning the need for delivery to the laboratory or need for analysis.

Analysis Completeness.

The analysis completeness will be (a) the number of laboratory samples for which actual valid results are reported compared to (b) the number of laboratory samples delivered to the respective laboratory. Special considerations/computations will be made for sample splitting or compositing.

In all individual cases, completeness can also be expressed as a

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percentage. Further, for each of the three types of completeness, overall average values will be computed.

Appendix D

Letters of Instruction - Dugway Proving Ground
(This is a list only. The complete LOIs are in the TDP.)

LOI 1, Rev. 6 BangBox Test Operations
LOI 2, Rev. 1 Instruments and Calibration: Continuous
Analyzers and Discrete Samplers
LOI 3, Rev. 1 Data Acquisition System

Appendix E

Letters of Instruction - ECO, L.C.
(This is a list only. The complete LOIs are in the TDP.)

LOI 29 Bang Box Explosive Detonation and Munitions
Destruction Trials
LOI 30 BangBox Detonating Cord Detonation Trials
LOI 31 BangBox Low-density Tritonal Detonation Trials
LOI 32 BangBox Low-density Tritonal with additives
Detonation Trials
LOI 33 BangBox Low-density Amatol Detonation Trials
LOI 34 BangBox Low-density RDX/TNT/Wax Detonation Trials
LOI 35 BangBox Low-density RDX/TNT/Al/Wax Detonation Trials
LOI 36 BangBox Low-density Tritonal With Water Detonation
Trials
LOI 37 BangBox Low-density Amatol With Water Detonation
Trials
LOI 38 Aluminized Composite Propellant With Waste Burn
Tests

Appendix F

Letters of Instruction - MHW Laboratory
(This is a list only. The complete LOIs are in the TDP.)

CH Analytical Method, Standard Procedure for the
Micro-Determination of Carbon and Hydrogen
N Analytical Method, Standard Modified Dumas
Procedure for the Micro-Determination of
Nitrogen

Appendix G

Letters of Instruction - Mountain States Analytical, Incorporated (This is a list only. The complete LOIs are in the TDP.)

- LOI 001 Handling of Data
- LOI 002 Preparation, Handling, and Storage of Quartz Fiber Filters and Filter Samples
- LOI 003 Cleaning, Handling, Storage, and Use of Burn Pans, Wire Brushes, and Tote Boxes
- LOI 004 Collection, Handling, and Storage of Burn Pan Residues
- LOI 005 Extraction of Quartz Fiber Filter Samples for SFC/MS and SFC/TEA and Handling of Extracts
- LOI 006 Extraction of Quartz Fiber Filter Samples for GC/MS and Handling of Extracts
- LOI 007 Extraction of Burn Pan Residue Samples for SFC/MS and SFC/TEA and Handling of Extracts
- LOI 008 Extraction of Burn Pan Residue Samples for GC/MS and Handling of Extracts
- LOI 009 Soxhlet Extractor Procedure
- LOI 010 Concentration by Rotary Evaporation
- LOI 011 Supercritical Fluid Chromatography/Mass Spectrometry (SFC/MS)
- LOI 012 Supercritical Fluid Chromatography/Chemiluminescence Detection (SFC/TEA)
- LOI 013 Gas Chromatography/Mass Spectrometry (GC/MS)
- LOI 014 Semivolatile Organic Target Analyte Selection
- LOI 015 Sampling and Determination of Particulate Matter as PM₁₀
- LOI 016 Digestion of Quartz Fiber Filter Samples for ICP
- LOI 017 Digestion of Quartz Fiber Filter Samples for CVAA
- LOI 018 Inductively Coupled Plasma Optical Emission
- LOI 019 Cold Vapor Atomic Absorption Spectrometry
- LOI 020 Cleaning and Handling of Summa Canisters
- LOI 021 Collection of Air Samples with Summa Canisters
- LOI 022 Determination of Sulfur Hexafluoride (SF₆) in Summa Canister Samples
- LOI 023 Determination of Non-methane Organic Compounds in Summa Canister Samples
- LOI 024 Determination of Volatile Organic Compounds in Summa Canister Samples
- LOI 025 Determination of Fixed Gases in Summa Canister Samples
- LOI 026 Quality Control Plan: Procedures for Accuracy, Precision, and Completeness

Appendix H

Letter of Instruction - Oregon Graduate Institute
(This is a list only. The complete LOIs are in the TDP.)

LOI (September 18, 1995) Standard Operating Procedures, OB/OD
Dugway, Utah

Appendix I

Letters of Instruction - Radian Corporation
(This is a list only. The complete LOIs are in the TDP.)

LOI 10, Rev. 1 Quality Assurance/Quality Control Procedures
for Accuracy, Precision, and Completeness
LOI 26, Rev. 2 SVOC Sampling Procedure
LOI 27, Rev. 1 XAD-2 Preparation
LOI 28, Rev. 1 Sample Preparation
LOI 29, Rev. 1 Gas Chromatography/Mass Spectrometry (GC/MS)
LOI 32, Rev. 1 Sample Custody and Identification
LOI 33, Rev. 1 Dioxin and Furan Sampling and Analysis
LOI 39 Determination of Hydrogen Chloride Sampling
Procedure

Appendix J

Letters of Instruction - Sunset Laboratories
(The complete LOIs are in the TDP.)

Sunset Laboratory Thermal-Optical Analysis for
Organic/Elemental Carbon Aerosol

**Appendix K Hazardous and Solid Waste Management Annex for BangBox
Testing, U. S. Army Dugway Proving Ground (Reference only.
Complete copy is in the TDP.)**

**Appendix L Standing Operating Procedures for: Propellant,
Explosive, and Pyrotechnic Thermal Treatment Evaluation Test
Facility (PEP-TTET) (BangBox) (Reference only. Complete copy is
in the TDP.)**

**Appendix M Safety Annex for Open Burning/Open Detonation (OB/OD)
Testing Within the Combustion Products Test Facility (Reference
only. Complete copy is in the TDP.)**

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APPENDIX H. LETTERS OF INSTRUCTION

WEST DESERT TEST CENTER

Title	LOI Number
BangBox Test Operations	1
Instruments and Calibration: Continuous Analyzers and Discrete Samplers	2
Data Acquisition System	3
BangBox Explosive Detonation and Munitions Destruction Trials	29
BangBox Detonating Cord Detonation Trials	30
BangBox Low-Density Tritonal Detonation Trials	31
BangBox Low-Density Tritonal with Additives Detonation Trials	32
BangBox Low-Density Amatol Detonation Trials	33
BangBox Low-Density RDX/TNT/Wax Detonation Trials	34
BangBox Low-Density RDX/TNT/Aluminum/Wax Detonation Trials	35
BangBox Low-Density Tritonal with Water Detonation Trials	36
BangBox Low-Density Amatol with Water Detonation Trials	37
Aluminized Composite Propellant with Waste Burn Trials	40
Diesel Fuel/Dunnage Waste Burn Trials	41

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1. BANGBOX TEST OPERATIONS

This letter of instruction outlines the BangBox (BB) test operations procedures that will be followed as a part of the BangBox test program at the U.S. Army Dugway Proving Ground (DPG). Facility requirements for test preparation, test procedures, and sequences of events are included.

2. TESTING DESCRIPTION

A series of tests will be carried out at DPG that will involve the detonation of explosive munitions and the burning of propellants. The Propellant, Explosive, and Pyrotechnic Thermal Treatment Evaluation and Test Facility, commonly known as the BangBox, will be used to contain the detonation or combustion cloud of test munitions in order to collect both gaseous and aerosol samples so that a complete characterization of their emission products can be accomplished.

3. BB TEST FACILITY STRUCTURE

Standing operating procedure (SOP) for the BB is contained in West Desert Test Center, Dugway Proving Ground SOP Number DP-000-P-851 with Change number 1 dated 4 October 1994. All personnel participating in BB tests are required to read and sign the command post (CP) copy of the SOP prior to test operations. This standing procedure complies with DPG and Department of the Army policies and procedures for test facilities and includes safety, security, and environmental hygiene issues. The BB SOP also details the operation and controls for the inflation system, which is the central system of the structure function.

3.1. Test Chamber

The BB test chamber wall is a flexible polyvinylchloride (PVC)-coated polyester fabric in the shape of a hemisphere sixteen meters in diameter. The chamber volume is maintained at approximately 1008 cubic meters by means of the BB inflation system which provides air pressure in the range of +1.1 to +1.5 inches of water column (in.w.c.) differential to ambient air pressure. The chamber serves to capture the clouds of detonation and/or burning of energetic materials being tested. The captured cloud is contained in the chamber, diluted at a controlled rate, and sampled over the period designated in the detailed test plan (DTP). The sampling period normally extends from 35 to 60 min.

The number of personnel in the test chamber prior to tests and between test trials must be kept to the minimum required to accomplish designated tasks, and then only for as long as necessary. Purging of the chamber atmosphere after personnel leave may be required to achieve 'background' conditions, especially for carbon dioxide (CO₂) gas.

Discrete samplers are placed in the test chamber for collection of particulate matter for laboratory analyses. Samplers to be used are outlined in the test design test plan. All sampling equipment must be ready for test approximately 30 minutes prior to ignition or detonation.

3.2. Inflation System

The BB inflation system provides ambient air to the test chamber to maintain structure support and volume. The system is comprised of a primary and secondary blower and an emergency gasoline powered engine. The primary blower operates at all times. The secondary blower is activated in the event the primary blower is unable to maintain +0.75 in.w.c.differential to ambient air pressure. It is deactivated when the differential pressure achieves +1.0 in.w.c.. During periods of unsettled ambient conditions, such as gusting winds, the secondary blower is placed in the 'firm' position to operate simultaneous with the primary unit. The secondary blower can also operate in the 'firm' position to provide air pressure in the event of failure of the primary unit. The emergency engine is gasoline (unleaded) powered and activates in the event of loss of electrical power. It starts automatically upon power loss by a twelve volt battery powered starting motor and provides rotation of the secondary blower fan through a mechanical power linkage. The secondary blower then provides air pressure to the BB test chamber.

The operation of the inflation system controls and the emergency engine are tested every working day, including test days. Entries are made accordingly in the BB log, including observations regarding potential malfunction or requiring immediate maintenance.

In the event of loss of electric power service to the BB facility during tests, test personnel will stand clear of the facility to allow orderly operation of the emergency engine and controlled deflation of the test chamber if necessary.

3.3. Airlock

The BB airlock is an interlock passageway to achieve access to the test chamber in such a way as to minimize air pressure loss from the test chamber. This is accomplished by ensuring that no

more than one inside or outside personnel or overhead door are open at the same time. All doors remain closed except for ingress or egress.

The BB airlock also serves as the sampling and continuous recording analyzers center. Continuous analyzers for criteria pollutants, toxic gases, PM₁₀ particulate, and discrete sampling in canisters are connected to the sampling manifold. Electric inputs to the data acquisition system provide information which is converted into useful data and engineering units.

The airlock has a pressurization system that will be activated during sampling operations. This system provides sufficient internal air pressure to prevent sample gases under pressure in the test chamber to leak into the airlock and contaminate instrumentation sampling processes or test personnel. The blower will be turned off during non-test conditions.

3.4. Burn Pad

The burn pad is located in the center of the BB test chamber and is constructed of steel plate. It is one square meter and lies flat on the floor.

Burning of test material is conducted by placing the material in a stainless steel burn pan and putting it on the burn pad. Preparation of test materials is performed only by qualified munitions personnel. Charges are weighed prior to ignition and the weight recorded. Munitions personnel are the last persons in the test chamber prior to firing, and the first to re-enter after the sampling period. They are responsible for the safety of ignition/detonation operations and insure the BB is evacuated before firing the test trial and that all is safe again prior to personnel re-entry of the test chamber.

After completion of each test trial the floor of the suppressive shield will be swept clean of residues using a foxtail brush and dust pan. Residue samples are then collected for analysis and the BB structure fabric and floor will washed, cleaned, and made ready for the next test.

3.5. Overpressure Hatch

The hatch is a weighted trap door located in the overhead of the test chamber entryway which opens when a rapid overpressure occurs. This serves to protect the BB structure from stress rupture. Weights are available to minimize gaseous release by instantly returning the hatch to a sealed position prior to cloud mixing and commencement of sampling.

The hatch is also used to provide a passage to purge the test chamber of sample gases. This will be used whenever necessary during post test procedures to render the test chamber safe for re-entry. It may also provide rapid purge prior to sampling to bring CO₂ concentrations down to background levels after test personnel complete sampler setup and exit the test chamber.

4. TEST EQUIPMENT

Test equipment requirements are established in the DTP. Analyzers, samplers, calibrators and support equipment will be ready in all respects to operate and set in place to comply.

4.1. Sampling Manifold

The stainless steel, polished manifold provides sampling ports for gaseous and discrete (canister) samplers. The manifold is configured to sample from both inside the test chamber and ambient (outside). It is internally swabbed clean with distilled and deionized pure water, then purged of residual gases with a nitrogen purge, and leak tested prior to sampling.

4.2. Real Time Analyzers

Calibration, sampling, maintenance, and quality control procedures for real time analyzers are contained in West Desert Test Center (WDTC) LOI 2.

4.3. Aerosol Samples

Samples are extracted to canisters from valve ports in the sample manifold. Specific requirements are contained in the DTP.

4.4. Particulate Samplers

Continuous monitoring and discrete sampling of particulate will be conducted. Total sample particulate (TSP) will be collected on specified media by high volume and PS1 samplers. Particulate measuring in the particle size range of 0-10 microns (PM₁₀) is monitored for concentration and weight and PM10 particulate samples collected for analyses as contained in the DTP. West Desert Test Center LOI 2 contains calibration and sampling procedures.

4.5. Air Circulator Fans

High and low speed industrial air circulators will be operated in the test chamber. Fan locations and direction of flow are configured to provide maximum mixing of burning clouds in the BB test chamber. The BB facility manager and/or BangBox Test Director (TD) will ensure that fans are operating prior to personnel exit from the BB during sampling..

4.6. Heaters

Salamander type space heaters (electric) are located in the test chamber. These will be operated for temperature control or removed as necessary to support the DTP requirements.

4.7. Vacuum Pumps

Vacuum pumps are used for providing sample flow through the sample manifold, pressurized flow to the 'zero' air generation system, and sample flow to the PM10 monitoring system. Flow volumes through these systems must be verified prior to and after testing with calibrated dry gas meters. Before and after test flow rate are compared to ascertain continuity and identify potential flow corrections to data.

4.8. Environment Instruments

These instruments provide measurements necessary to reduce data to standard conditions and units. Temperature, barometric pressure in the test chamber, relative humidity, and test chamber differential pressure will be recorded. Data is recorded in the data acquisition system (DAS).

5. DATA ACQUISITION SYSTEM

The DAS collects real time data for continuous analyzers, environmental measurements, samplers using flow devices, and other test inputs. The DAS is described in detail in WDTC LOI 3.

6. DETONATION/IGNITION FIRING SYSTEM (DIFS)

The DIFS provides electric current for activation of ignition devices used to detonate and/or burn test munitions. This system is operated only by qualified munitions personnel and is the key element to safe operations. Separate LOIs describe procedures for DIFS operations

7. COMMAND POST

The CP trailer is located approximately 540 meters from the BB. It contains the DAS file server, remote DAS monitors, the DIFS firing station, and a small work station for conducting test support. All personnel report to the CP prior to trial ignition and remain during sampling operations. Only necessary sampling and DAS test personnel are authorized to be in the BB airlock during sampling. The TD will monitor all test operations.

8. TEST MATERIALS

8.1. Portable Magazine

A portable, skid mounted munitions storage magazine for holding munitions and donor charge material for the days testing is located approximately 500 m north of the BB.

8.2. Munitions Preparation Trailer

The munitions preparation trailer is used to weigh test materials and prepare the munitions and donor material prior to placement in the BB suppressive shield detonation/ignition chamber.

9. SAMPLE ANALYSES

Sample analyses will be conducted to meet the requirements of the DTP. Real time analyzers, environmental measurement sensors, high volume and other flow devices provide input to the DAS for analysis. Discrete samples in canisters, on filter media, and residues will be submitted for laboratory analysis as specified by the DTP.

10. TEST OUTLINE

10.1. Sequence of Events for Sampling

A complete characterization test for each munition consists of an ambient background sample and three trials with each trial providing independent samples. The sequence of events is the same for both, with the exception that the background is without detonation or ignition of test materials.

10.2. Background Sampling

The same test procedures that apply to test material trials apply to background tests. Preparation of test equipment and test materials are contained in specific LOIs, DPG SOPs, and in the DTP.

10.3. Sequence of Events for Testing

Test preparations will be complete and the test facility ready approximately 60 minutes prior to ignition/sampling times. A typical test day would have trials as follows:

0730	BB test facility ready for test
0830	Begin test background sample
1030	Begin test trial #1
1230	Begin test trial #2
1430	Begin test trial #3
1530	Clean test facility for next test

Note: Some test materials may require cleaning the test chamber after every trial.

11. MASTER SEQUENCE OF EVENTS

11.1. Preparation Phase

The first test of any given test day is normally for background data to use as a baseline during data analysis. All required preparation items must be in place approximately 30 min prior to beginning data collection for backgrounds, or detonation/ignition of munitions test items.

Preparation items that require lead or long lead times to accomplish must commence in sufficient time to meet this schedule. Responsibility for completion of preparation items is as specified, or reasonably implied by the DTP.

11.2. Pre-test

The following items must be accomplished prior to being 'ready' for testing:

- Sampling materials, including travel blanks, on site at BB
- Instrument daily zero and span calibration checks complete

- All operators read and sign the current DPG BB SOP DP-000-P-851
- Conduct daily safety briefing for all test personnel
- Munitions designated for the test period are available in the portable munitions magazine
- Specified scales and paper sacks are available in the munitions preparation trailer
- Medical services personnel are on site or standing by at Ditto Aid Station
- Clear area - all non-test personnel depart BB and report to TD at CP
- Close Prime Road to non-test traffic - place standard barricades at intersection of Prime Road and Stark Road and 300 yards north of the portable magazine
- Notify DPG Range Control office of road closure and BB test facility going 'hot'
- Proper fire symbol displayed while testing munitions items
- Red warning light ON when operating BB

11.3. Background Sample

Background samples are performed prior to test trials for each munition. The background test is performed with the same instrument and sampler requirements as a live test trial. The sample gas will be drawn from the sample manifold alternating between outside ambient source and inside test chamber source at five minute intervals. Background sampling will be conducted for a minimum of 35 min.

11.4. Test

Where there is conflict between instructions, provisions of the DTP will govern.

The list of events and times are subject to change before detonation or ignition. 'T' is the symbol that refers to the event of detonation or ignition. Times are given in minutes from this reference point.

The BangBox Sampling Checklist is found in Appendix B. Event times will be recorded accurately using the time as displayed on the DAS monitor.

Appendix C is a form to record times of significant events of interest for data reduction and analysis and quality control purposes for background and trial sampling.

The following is a typical series of events performed in a test sequence.

Prior to T-60	Analyzers and samplers and data acquisition systems ready in all respects
T-60 to T-15	Install sample media Dry ice and coolers on site Expendable items in place (filters, sample bottles, burn pans, etc.) Check DAS display for anomalies Check all vacuum pumps for performance Sample line heater(s) operating Prepare munitions for setup in BB Brief BB visitors at BB Real-time analyzers apparatus/flow checks Sample line scrubbers in place and ready
T-15 to T-5	Place munitions in BB Munitions personnel complete material setup and exit test chamber Purge BB to ambient gas levels
T-5 to T	Close overpressure door (make sure proper weights in place) Check closure of all doors Start Hi-Vols, PS1s, HiVol-PM-10s, circulating fans, heaters (if required) Collect 6-L and 0.85-L samples Record final background on DAS Turn airlock pressurization system 'on' Assure red light at BB is ON and flashing Relocate all personnel to CP Check that road barricade and placard are displayed
T	Detonation or Ignition from CP
T+5	Sample VOCs (inside and out), and SF ₆
T+7	Sample SF ₆
T+11	Sample SF ₆

T+19 Sample SF₆

T+35 Sample SF₆

T+36 End of trial
 Shut sampling off
 Weapons personnel enter BB when safe gas level achieved

T+37 Start purge of chamber to safe gas levels for entry
 Weapons personnel declare BB entry permitted
 Turn airlock pressurization system 'off'
 Sampling crew collect samples, check labeling and prepare for shipment to
 laboratories designated for analysis.

11.5. Post-test

Clean BB test chamber
Open Prime Road by removing barricade
Notify Range Control when the test facility is 'cold'
Turn OFF red flashing light
Release medical personnel from the test site
Remove fire symbols from test facility
Complete logbook entries with date and signatures on each page
Ensure BB and CP equipment is secure and doors locked
Samples not shipped place in walk-in freezer at work center

APPENDIX A

BANGBOX SAMPLING PREPARATION CHECK LIST

Date: _____

Item: _____

Instrument or Sampler	Calibration multiple pt	'Zero'/Span	Setup/Pre-Operation	DAS Setup/Operating	Valves in Position	Filters in Place
CO ₂						
CO						
SO ₂						
NO-NO ₂ -NO _x						
O ₃						
HCl						NA
MDA ()						NA
PM ₁₀						
SF ₆ INST						NA
HI-VOLsSFC					NA	
HI-VOLsGC					NA	
HI-VOL MET					NA	
HI-VOL PM ₁₀					NA	
HI-VOL 1B					NA	
HI-VOLs EX					NA	
PS1s						
CANISTERs	NA	NA		NA		NA
MIDG IMPIN				NA	NA	NA
RTDs		NA			NA	NA
BARO.		NA			NA	NA

RH		NA			NA	NA
ΔPs		NA			NA	NA

APPENDIX B

BANGBOX SAMPLING CHECK LIST

Date: _____

Item: _____

I.D. : _____

TIME	EVENT READY		TIME	EVENT
	Test Chamber; clean, setup, OPH open			TEST IN PROCESS LIGHT ON
	Circulating fan #1 on			BEGIN SAMPLING : All samplers
	Sample valves open, calibration closed			MDA Instrument to 'Analysis'
	Real-time instrument suite			DAS : All systems OP ()
	Ignition check			Canister Background Sample
	Real-time (SF ₆)			Masks and Drager kit in place
	Real-time (HCl & Cl ₂ Midget Impinger)			Airlock overpressure pump on
	Hi-vols; SFC/MS, GC/MS, Metals, PM ₁₀			EXIT Airlock
	Hi-vols; (Extraction method test)			Explosives sign in place
	PS1s; GC/MS			PS1 exhaust conduit cap off
	PS1 Dioxins			Shunt (WEAPONS ONLY)
	Canister System (SF ₆ , TOCs, GC/MS)			IGNITION/DETONATION
	Canister System (Multiple locations)			Airlock entry (HCl Inst. ____ppm)
	TC conduit cap on			HCl(____ppm),Cl ₂ (____ppm)-Drager
	All circulating fans on			STOP SAMPLING
	Sample manifold pump on,			Canister Sampling complete
	Sample line heater on : HCl Instrument			Enter TC, (HCl Inst.____ppm)
	BB clear of non-essential personnel			Open OPD (hatch) & Exit TC
	SF ₆ Release set			Enter TC, (HCl Inst ____ppm)

	Test material setup complete, TC clear			Drager HCl___ppm, Cl ₂ ___ppm
	OVERPRESSURE HATCH CLOSED			TC cleared for personnel entry
	Acquisition System GO			Fan #1 on
	Nitrogen purge/CO ₂ Instrument(ppm)			TEST IN PROCESS LIGHT OFF

APPENDIX C

SAMPLING SCHEDULE/REMARKS

DATE : _____

TEST ITEM : _____

BACKGROUND SAMPLE

TIME
SOURCE OF SAMPLE

_____ BEGIN
OUTSIDE BANGBOX

_____ INSIDE TEST CHAMBER

_____ OUTSIDE BANGBOX

_____ INSIDE TEST CHAMBER

_____ OUTSIDE BANGBOX

_____ INSIDE TEST CHAMBER

_____ OUTSIDE BANGBOX

STOP

_____ PUT VALVE TO INSIDE TC FOR TEST TRIALS

COMMENTS

TRIAL #1

TRIAL #2

TRIAL #3

1. INSTRUMENTS AND CALIBRATION: CONTINUOUS ANALYZERS AND DISCRETE SAMPLERS

This letter of instruction specifies the BangBox (BB) test facility instrument suite for test operations. It includes calibration, sampling, maintenance and quality control procedures that will be followed as a part of the BangBox test program at the U.S. Army Dugway Proving Ground (DPG).

2. TESTING DESCRIPTION

A series of tests will be carried out at DPG that will involve the detonation of explosive munitions and the burning of propellants. The Propellant, Explosive, and Pyrotechnic Thermal Treatment Evaluation and Test Facility, commonly known as the BangBox will be used to contain the detonation or combustion cloud in order to collect both gaseous and aerosol samples so that a complete characterization of the emission products can be accomplished. The detailed test plan (DTP) for each test series provides specific requirements for the sampling suite, itemizes test materials, and gives instructions on the disposition of discrete samples for distribution to commercial laboratories for sample analysis.

3. INSTRUMENT SUITE

The suite of instruments for BB tests includes real time instruments, discrete samplers, and environmental condition sensors/transmitters located in the facility airlock. A sample manifold provides sample gases for real time instruments and sampling canisters as outlined in the DTP.

3.1. Real Time Instruments

Continuous gas analyzers and particulate monitors provide real time voltage data to the data acquisition system (DAS) for recording. These instruments detect concentrations of gases for CO, CO₂, NO-NO₂-NO_x, SO₂, O₃, HCl, Cl₂/HCN, SF₆, and particulate data for PM₁₀. These instruments are designed for continuous operation and will be turned ON a minimum of 12 hr prior to tests.

3.2. Discrete Samplers

Discrete samplers located in the test chamber include high-volume total sample particulate (TSP) samplers for collecting aerosol particulate on 20.3 cm x 25.4 cm quartz fiber filters. A high volume particulate sampler with a specially designed inlet for separating particle sizes with average diameter of 10 μm or less (PM10) and pesticide particulate and vapor collection samplers which collect particulate on a 100 mm diameter quartz fiber filter and XAD-2TM resin cartridge using General Metal Works (GMW) PS1 samplers. These are also positioned in the BB test chamber. All flow producing samplers provide current data to the DAS through transmitters for continuous flow calculations.

A USEPA Method 26 dual train impinger mini-sampler is used to collect gaseous samples for HCl and Cl₂ analyses.

Other discrete sampling devices are located in the facility airlock to collect aerosol samples in 6-L and 0.85-L canisters from the sampling manifold for further analysis.

4. ENVIRONMENT

Environmental conditions are monitored with sensors for temperature, test chamber and air lock differential pressures, relative humidity, and barometric pressure. This data is monitored and recorded continuously and used to calculate concentrations and volumes in standard units.

5. CALIBRATION

5.1. Basis

Calibration of instruments will follow procedures detailed by instrument manufacturers operations manuals and technical data sheets as supplemented by instructions provided by U.S. Environmental Protection Agency (USEPA) quality assurance/quality control (QA/QC) audit personnel. Copies of manuals and instruction sheets will be available in the BB airlock for reference at all times.

5.2. Gases

The 'zero-air' generation system and gas dilution system will interface with real-time instruments through valve arrangements and provide gases for dynamic calibrations. Calibration gases for

real time gas analyzers of criteria pollutants are certified USEPA protocol gases (AREAL Dec 1993 Standard).

5.3. Discrete Samplers

Discrete samplers will be calibrated using devices specified, approved, and certified by sampler manufacturers and as modified by instructions provided by USEPA continuous emission monitor auditors. Calibration kits and instructions will be available for reference in the BB airlock at all times. Calibration curve coefficients will be developed using the ReFlows calculation formulations in Lotus 1-2-3 and regression analysis to provide calibration curve fit for DAS data reduction to voltage and engineering units.

6. BB DATA ACQUISITION SYSTEM

The DAS operation, calibration, and data filing procedures that will be carried out as a part of the test program at the West Desert Test Center are contained in West Desert Test Center letter of instruction (LOI) 3. A description of hardware/software components, wiring, data storage formats, calibration and operation procedures are included, providing a detailed instructional guideline of the steps that will be used to gather and validate data taken from test equipment.

7. SAMPLE ANALYSIS

Sample analyses will be conducted to meet the requirements of the DTP. Real time analyzers, environmental measurement sensors, high volume and other flow devices provide input to the DAS for analysis. Discrete samples in canisters, on filter media, bubbler/scrubbing devices, and residues will be submitted for laboratory analysis as specified by the DTP.

8. QUALITY ASSURANCE/QUALITY CONTROL

8.1. Oversight and Monitoring

Quality assurance and quality control principles and procedures will be followed during all operations of BB tests. All BB facility and test personnel will comply with requirements as outlined and specified in standing operations procedures, DTP, and LOI. The BangBox Test Director will direct QA oversight and monitoring of BB operations and test personnel.

8.2. Chain-of-Custody

Laboratory personnel on site retain custody of sample materials produced in their area of responsibility, as assigned by the DTP. All BB test personnel with sampling responsibility will transfer custody to designated laboratories for assay at the earliest possible opportunity. Sample custody records will be strictly maintained.

9. INSTRUMENT DATA

9.1. NO_x Instrument

Separate multi-point calibrations will be done on the NO and NO₂ channels of the instrument using a National Institute of Science and Technology (NIST) traceable, U.S. Environmental Protection Agency (EPA) Protocol (December 1993) NO test gas at the start and completion of the test series. This gas will be diluted to the working range of the instrument using a gas dilution system that includes calibrant gas pressure flow control and a zero air supply for diluent gas. Five gas concentration levels will be generated and sampled with the instrument. The voltage response of the instrument is determined for each concentration level. The sample gas will be delivered to the instrument through a Teflon™ in-line filter installed on the instrument inlet. The conversion factors for the determination of the concentration levels from voltage output from the instrument will be determined by linear regression of the input concentration of the test gas against the measured voltage output for all test concentrations generated. The calibration response factors will be considered valid if the correlation coefficient (r^2) of the regression is greater than 0.98. All calibration data will be recorded in the instrument logbook.

Daily checks on instrument performance will be carried out using a zero gas and a NO span gas with a concentration in the normal working range of the instrument. In the event that the recorded instrument output during a span gas check deviates from the expected output by more than ± 10 percent, the instrument will be checked and the problem remedied. Following any significant repairs on the instrument, a multi-point calibration will be carried out prior to placement of the instrument back in service. Results from all daily zero and span checks will be recorded in the instrument logbook.

9.2. CO Instrument

Multi-point calibrations comparable to those for the NO_x instrument will be performed on the CO instrument using a certified EPA Protocol CO test gas and a gas dilution system. Daily zero

and span checks will be carried out using a zero gas source and a working range CO span gas. A ± 10 percent accuracy criteria will be applied to the span check results to determine acceptable instrument performance on a daily basis. Results from all daily zero and span checks will be recorded in the instrument logbook.

9.3. CO₂ Instrument

Multi-point calibrations comparable to those for the NO_x instrument will be performed on the CO₂ instrument using a certified EPA Protocol CO₂ test gas and a gas dilution system.

Daily zero and span checks will be carried out using a zero gas source and a working range CO₂ span gas. A ± 10 percent accuracy criteria will be applied to the span check results to determine acceptable instrument performance on a daily basis. Results from all daily zero and span checks will be recorded in the instrument logbook.

9.4. SO₂ Instrument

Multi-point calibrations comparable to those for the NO_x instrument will be performed on the SO₂ instrument using a certified EPA Protocol SO₂ test gas and a gas dilution system.

Daily zero and span checks will be carried out using a zero gas source and a working range SO₂ span gas. A ± 10 percent accuracy criteria will be applied to the span check results to determine acceptable instrument performance on a daily basis. Results from zero and span checks will be recorded in the instrument logbook.

9.5. HCl Instrument

Multi-point calibrations comparable to those for the NO_x instrument will be performed on the HCl instrument using a certified CO₂ test gas and a gas dilution system. Daily zero and span checks will be carried out using a zero gas source and a working range HCl span gas. A ± 10 percent accuracy criteria will be applied to the span check results to determine acceptable instrument performance on a daily basis. Results from all daily zero and span checks will be recorded in the instrument logbook.

Due to the nature of HCl, a separate heated sampling line will be provided to this instrument when test materials contain compounds with Chlorine. Concurrently, a chlorine scrubber will be

attached to the sample manifold inlet to prevent potential chlorine contamination in the sample manifold and in other instruments.

9.6. O₃ Instrument

A multi-point calibration of the O₃ instrument will be conducted prior to the test series using a certified ozone generator. The calibration gas will be passed through an in-line Teflon™ filter connected to the inlet of the instrument.

Daily zero checks will be carried out on the instrument prior to each test. Results from all daily zero and span checks will be recorded in the instrument logbook.

9.7. Cl₂/HCN/Instrument

The MDA Scientific, Inc. Series 7100 Continuous Toxic Gas Monitor is used for analysis of HCN during normal characterization sampling and for Cl₂ when sampling test materials containing chlorine. This instrument uses an optics calibration stain card that is directly traceable to the original dynamic calibration and will be calibrated according to manufacturer instructions.

Due to the nature of HCl, a separate sampling line will be provided to this instrument when test materials contain compounds with Chlorine. Concurrently, a chlorine scrubber will be attached to the sample manifold inlet to prevent potential chlorine contamination in the sample manifold and in other instruments.

9.8. Sulfur Hexafluoride (SF₆) Continuous Analyzer

The operation and calibration of the SF₆ continuous analyzer are contained in a separate LOI.

9.9. PM10 Particulate Monitor

The Rupprecht & Patashnick Co., Inc TEOM® Series 1400a Ambient Particulate (PM10) Monitor provides a continuous output to the DAS for concentrations of particulate in the sample cloud as well as mass accumulation over a sample period. The particulate identified is the total mass of those particles that have an average diameter of ten micrometers and less. Calibration of this instrument is in accordance with the manufacturer instructions in the instrument operating manual.

9.10. High-Volume TSP and PM10 Samplers

The quartz fiber filters taken during background and test cloud sampling by high volume and PM₁₀ samplers will be submitted to the assigned laboratory for assay as specified in the DTP. The filters will first be weighed to determine the baseline for calculation of aerosol weight collected. Detailed analytical procedures are given for the laboratories in their respective LOI.

9.11. Pesticide Particulate and Vapor Collection System

The GMW Model PS1 Sampler uses XAD-2™ cartridges and quartz fiber filters for sample collection during background and test cloud sampling. Samples will be submitted to the assigned laboratories for assay of semi-volatile organics compounds (SVOC) and dioxins. Detailed analytical procedures are given in the respective laboratory LOIs.

9.12. USEPA Method 26

A USEPA Method 26 dual train impinger mini-sampler is used to collect gaseous samples for HCl and Cl₂ analyses. Operating and calibration procedures are contained in a separate LOI.

9.13. 6-L Canisters

Aerosol samples will be taken from the sample manifold at intervals specified in DPG LOI 1. The sample filled 6-L SUMMA™ canisters will be submitted to assigned laboratories for VOC assay. Complete details of this procedure are given in the LOIs provided by each laboratory providing sampling and analysis.

9.14. Sulfur Hexafluoride (SF₆) Release System

This system releases SF₆ gas of a known quantity and concentration in the BB test chamber at the time of test ignition. Procedures are as outline in the DTP and laboratory LOI.

9.15. 0.850-L Canisters

Aerosol samples will be taken from both the sample manifold and a separate sampling system from locations and at intervals specified in the DTP. The sample filled 0.850-L canisters will be submitted to the assigned laboratory for assay of SF₆.

9.16. Air Flow Meters

Dry test meters will be checked and certified for flow prior to the onset of test. Flow measurements of sampling instruments and sample manifolds will be conducted prior to and after each test series to verify flow rates and instrument concentration outputs. Prior to initial flow tests the sampling lines for each system will be purged with high purity nitrogen gas and leak tested.

10. SAMPLING PROCEDURES

10.1. Filter Handling

Filter handling procedures are detailed in LOIs prepared by laboratories as specified in the DTP. Test personnel who may assist laboratory personnel in the handling of filters must first become well versed in the LOI pertaining to the specific laboratory responsible for the particular discrete samples, and then may assist only with the concurrent authorization of the TD and senior team member on site representing the responsible laboratory.

10.2. High-Volume Sampler Filter Loading

Prior to loading the filters in a high-volume sampler, the filter housings will be cleaned with reagent grade isopropanol and tissue. Personnel loading the filters will wear clean nylon gloves to minimize cross contamination. The filters will be removed from their glass bottles and placed on the backup screen. An isopropanol-wiped foam gasket is then placed on top of the filter, followed by the top half of the filter housing. The lock nuts are then snugged down such that good contact between the gasket and the two halves of the filter holders results. The filter number and unit position are then recorded on the labels and in the custody log.

10.3. High-Volume Sampler Filter Unloading

The above sequence is followed in reverse order for filter unloading. Following removal of the top half of the filter unit, the filter is folded in half and then in half again in order to minimize the loss of particulate material from the surface of the filter. The folded filter is inserted in its glass bottle, sealed with the Teflon™ lined lid and immediately placed in cold (dry ice) storage and held there until it is released to the analytical laboratory.

10.4. Burn Pans

Residue from the burning of propellants are collected from the propellant burn pans and placed in sample bottles. Residues gleaned from the floor of the suppressive shield are also collected for further analysis. Sampling procedures are outlined in the DTP and designated laboratory LOI.

1. DATA ACQUISITION SYSTEM

This document outlines the data acquisition system (DAS) operation, calibration, and data filing procedures that will be carried out as a part of the BangBox test program at Dugway Proving Ground (DPG). A description of hardware/software components, wiring, data storage formats, calibration and operation procedures are included, providing a detailed instructional guideline of the steps that will be used to gather and validate data taken from the test instrument suite.

2. TESTING DESCRIPTION

A series of tests will be carried out at DPG that will involve the burning of propellants and detonation of explosive munitions. The Propellant, Explosive, and Pyrotechnic Thermal Treatment Evaluation and Test facility, commonly known as the BangBox (BB), will be used to contain the detonation or combustion cloud in order to collect both gaseous and aerosol samples so that a complete characterization of the emission products can be accomplished. The BB DAS collects real-time data from continuous gas and particulate analyzers, sample gas flow data from discrete gas and particulate samplers, and pertinent environmental sensor information.

3. COMPONENT DESCRIPTION AND INTERFACE

3.1. Basic System Elements

Five computer systems connected to a BangBox local area network (LAN) comprise the basic system elements. Two computers located in the BangBox airlock provide data and video services to the LAN. The remaining three computers remotely stationed at the BangBox command post (CP) display or provide storage for real-time data generated at the BangBox. Of the remote computers, one system is configured solely as a file/application server. This machine is responsible for data and program storage, data backup, time synchronization, and master diagnostic console.

3.2. Data Acquisition System Hardware

The computer systems in the BB airlock and those located at the CP communicate by using fiber-optic cabling. A 10base-T hub at each location fitted with a 10base-FL transceiver provides a reliable high-speed data link. All data and video are passed digitally over the Ethernet link. In

the BangBox airlock an INTEL 80486DX/33 computer fitted with two Analog Devices acquisition cards and an IOTech DaqBook external acquisition device provide the interface to connect to the test instrumentation suite. The temperature, pressure, humidity, criteria pollutant instruments, toxic gas analyzers, and particulate samplers have data lines leading to this computer.

3.3. Voltage Output Instruments

Direct voltage outputs are provided by the continuous analyzers such as the criteria pollutant instruments, toxic gas analyzer, ambient PM₁₀ particulate monitor, and environmental pressure and barometric pressure transmitters. One of these circuits will be connected in parallel to the firing circuit to verify the presence or absence of an ignition current.

3.4. Data Acquisition System Software

3.4.1. Logging Software. LABTECH® NOTEBOOK version 8.03 will be the software responsible for polling each sampling channel at scheduled intervals, and for storing and transmitting this data.

3.4.2. Display Software. Data will be displayed using Lotus 1-2-3 Release 5 software with Network Dynamic Ddata Eexchange (NetDDE), an integral part of Microsoft Windows for Workgroups, providing the data transport mechanism.

4. OPERATION PROCEDURES

4.1. System Operation

Two days prior to testing, the system will be activated, which will generate data files containing one hour of data for each file. These raw data files will be stored on tape as part of the permanent record of operation. Acquisition will only be stopped prior to the end of the test series in the event of malfunction or miscalibration of test instruments requiring modification to the NOTEBOOK setup file. Acquisition will be resumed promptly following setup changes or hardware repairs.

4.2. Post-Test

A copy of the active configuration files will be placed with the data files for each trial. At the conclusion of each test day, files for that day will be copied to a removable disk cartridge for transport. The data stored on the file server will be maintained throughout the test cycle pending available disk space. At no time will data be deleted from this machine without concurrence of the DPG Test Director (TD) and with sufficient verified backups in place.

5. ACQUISITION SYSTEM CALIBRATION

5.1. Frequency

The performance of the data acquisition system will be checked against a voltage standard annually certified one month prior to the onset of the test.

5.2. Analog-to-Digital Conversion

Each data channel physically connected to a sensor or test instrument will be checked for voltage accuracy and linearity. The data acquisition system will be used as a voltage injector that will be attached to each channel in parallel with a digital volt meter (DVM) that has been calibrated and certified to meet manufacturer specification. Ten readings will be observed by the DVM and that of the acquisition system, then logged and entered into a regression table. Accuracy within a tolerance of plus or minus two percent of range and regression parameters with an R squared of 0.995 or greater will be acceptable.

5.3. System Integrity

Total system integrity will be checked by verification of proper voltage signals on the data files written by the system. In addition, time sequence marks will be validated to insure voltage signals are recorded with the proper time stamp and

6. DATA FILE FORMAT

For each trial and calibration sequence several data files will be generated and a current acquisition configuration will exist. Separate data files will be generated for each group of instruments that have a specific sample interval.

6.1. Sampling Interval

The DAS sampling interval will be set to match the corresponding instrument internal cycle. A typical 45 minute sampling time for each trial will contain approximately 270 data points per data channel. A one second interval will be used for instruments requiring higher resolution.

6.2. Data Files

Each data file will be arranged by row and column of ASCII formatted floating point numbers. Each row will contain all samples for a given time interval and each column will contain all data points for one specific acquisition channel. Each file will contain a header indicating when the file was initially created, the sampling interval, what the file represents, and what each column of data represents. Each column of data will represent the raw voltage data from a particular test instrument or sensor.

6.3. File Names

The file names will be generated automatically from the NOTEBOOK software using a sequence number to insure that no data files are created that matches a previous file name causing data to be overwritten.

6.4. Data Transfer

When data is transferred to removable cartridge disk, a directory structure will be created using Julian dates for names. All files belonging to a trial will be copied into the directory specified by the trial name.

1. BANGBOX EXPLOSIVE DETONATION AND MUNITIONS DESTRUCTION TRIALS

1.1. Munitions Designations for Trials

1.1.1. Explosives. The explosives to be detonated will be prepared as surrogates designed to represent munition loadings in the demilitarization inventory. Modifications to this LOI may be made if specific munitions are tested

1.1.2. Munitions for Burn Tests. Specific munitions previously detonated with a detonation/initiation train will be initiated and destroyed by exposing them to the flame of a natural gas or propane burner. The burner will be remotely actuated.

1.1.3. Blasting Caps. Exploding bridge wire blasting caps are most desirable for all munitions trials because of safety. However in the present trials, electric blasting caps will be used. When ignition only is necessary, electric squibs with additional powder may be used.

1.2. Support Material

1.2.1. Single Explosives.. Explosive charges will be detonated in the open in the BangBox. All charges will be contained in light polyethylene film packages and will be secured and mounted with non-metallic filament or string. The only metal present will be the blasting cap shell which will be consumed in the detonation. No other metal than the blasting cap leads will be used,

1.2.2. Explosive Charge Location. All explosive charges will be located at least 1 m above the floor of the BangBox, and centered over the firing pit cover. This cover may be covered by a metal plate to prevent the fireball from contacting the epoxy paint on the cover.

1.2.3. Mounting. The individual explosive charge will be suspended from a stand prepared from angle iron welded to a base plate, which plate will be inserted under the pit cover. The charge will be mounted horizontally by monofilament line or cotton string from the top ends of these angle iron stands.

1.3. Munition Burns

Selected munitions previously tested will be destroyed by heating over a burner placed in the lower part of the detonation firing pit. This simulates the destruction in the field by simple furnaces.

1.3.1. Multiple Munitions. Several pieces for each munition may be tested in the same trial as determined by the PEP content of the individual munition.

1.3.2. BangBox Protection. The BangBox will be protected from munition travel, when it is actuated by the flame, by placing at least one layer of an expanded metal screen over the top of the pit where the burner is located.

1.3.3. Burner Actuation. The burner in the pit will be remotely actuated including both the release of gas to the burner and the ignition of the flame.

2. EXPLOSIVE AND MUNITIONS PREPARATION

2.1. Identification

All explosives and munitions tested in the BangBox will be identified and all lot and other numbers will be recorded. This identification will also extend to aluminum, ammonium nitrate and other additives used in surrogate mixes.

2.2. Casing Removal

The munitions will be removed from the external casings or packaging before mounting for detonation trial. All packing material will be removed completely from the BangBox and disposed of in accordance with established DPG procedures.

2.3. Explosives and Munitions Handling

2.3.1. Personnel. All explosives and munitions will be handled only by qualified personnel.

2.3.2. Safety Devices. No munitions will be used unless all personnel protective devices are in place.

2.3.3. Initiation

2.3.3.1. Mode. Wherever practical the explosives and munitions will be initiated in the precise manner for which they were designed unless otherwise specified by the test officer. In no instance will the munitions be initiated in a manner which will endanger personnel.

2.3.3.2. Remote operation. All explosives and munitions will be remotely initiated.

2.3.3.3. Modification. Methods of initiation may be modified as needed to assure that the explosives and munitions will function correctly when remotely initiated.

2.3.3.4. Alternate methods. Munitions is to be tested by burning procedures will be tested under separate and distinct LOI.

3. MUNITIONS CONTAINING MORE THAN ONE EXPLOSIVE CLASS

3.1. Definition

These include any explosives and munitions where high explosives and/or propellants and/or pyrotechnics are present in the same munitions.

3.2. Safe Procedures

No munitions under the definition of 3.1 will be tested in such a manner that one or more of the materials will not be completely consumed.

4. INITIATION

4.1. Preparation for initiation

4.1.1. Procedures. Procedures will be established for each specific explosive or munition and approved by competent authority.

4.1.2. Personnel. Only competent designated personnel will arm charges. All other personnel will be out of the BangBox area before arming and will not return.

4.1.3. Interlocks. Firing circuits will have interlocks which keep firing lines short circuited and de-energized. This interlocks will only be opened at a remote location. Only personnel

responsible for firing circuit operation with have keys and authority to complete arming and firing of the munitions.

5. POST BANGBOX DETONATION PROCEDURES

5.1. Initiation Failure.

Failure to initiate will be treated as a misfire or unforeseen mishap and will be handled in accordance with DPG and WDTC standing operating procedures (SOP).

5.2. Inspection

Designated persons will inspect the BangBox after the detonation is completed in accordance with DPG and WDTC SOP. No other personnel will enter the BangBox until it is cleared.

5.3. Personnel Access

Further post detonation access to the BangBox will be as designated by the Test Officer.

6. ACCOUNTABILITY

6.1. Records

All pertinent data will be recorded for each test including explosive type, munitions type, method of support, method of initiation, and observations.

6.2. Compliance to LOI

Quality assurance/quality control procedures will be established to assure compliance with this LOI.

7. ADDITIONAL REFERENCES

West Desert Test Center and DPG references relating to the detonation of munitions modified as necessary for BangBox testing. Any instructions in conflict with these references will be resolved before proceeding with the test.

1. BANGBOX DETONATING CORD DETONATION TRIALS

1.1. Purpose

The purpose of these trials is to verify that the BangBox and its systems are ready for explosive testing, and to sample and analyze the emission products from the detonating cord.

1.2. BangBox Preparation

The detonating cord charge will be suspended at least 1-m above the center of the cover over the firing pit.

2. DETONATING CORD DESIGNATION

2.1. Explosive

The explosive will be 70 ft of detonating coiled in a loose spool.

2.2. Blasting caps

Exploding bridge wire blasting caps are preferable but such systems may not be available for the test. Electric blasting caps will be used instead.

2.3. Explosive Preparation

2.3.1. Measurement. At the preparation site, the detonating cord will be removed from the spool after verifying and recording the lot number and before preparing it for detonation.

2.3.2. Form of the Detonating-Cord Charge. The detonating cord will be formed into a loose coil and loosely secured with monofilament line or cotton string. The coil must not exceed 92 cm in diameter.

2.4. Support Material

2.4.1. Support Stands. Support stands will be placed opposite each other at the sides of the firing pit cover.

2.4.2. Charge Suspension. The charge will be suspended from the support stands with monofilament. The charge may be suspended vertically but the charge must be at least 15.2 cm above the cover.

3. CHARGE PLACEMENT

3.1. Personnel

The charge will be brought to the BangBox when ready for placement by qualified personnel only. All on-site mounting will be under their direction and in accordance with established procedures.

3.2. Detonating Cord Charge Placement

3.2.1. Time of Placement. The charge will be brought into the BangBox and mounted after all of the other preparations and checking of test equipment are completed, and personnel not needed to set the charge are evacuated.

3.2.2. Procedure. The charge will be suspended from the support stands as described in paragraph 2.4.2

3.2.3. Arming. The charge will be armed by fastening the cap in a small loop of detonating cord and securing it with at least one cable tie. Monofilament line may alternately be used but no soft iron or other metal line will be used to mount or arm the charge.

4. INITIATION OF DETONATION.

4.1. Responsible Personnel

Initiation of detonation will be by qualified personnel only. They will be responsible for personnel control, functioning of interlocks, warning and initiation of the charge.

4.2. Firing Circuit Operation

Firing circuit operation will follow procedures prepared separately and appropriate to this and all other LOI.

5. BANGBOX REENTRY

5.1. Airlock

As required and approved, limited test personnel may enter the air-lock adjacent to the BangBox to monitor instruments and obtain grab samples. They will not enter the BangBox itself until it has been cleared by the responsible weapons personnel.

5.2. Clearance

At the conclusion of the sampling period, responsible parties will enter the BangBox and inspect for an failures or unreacted detonating cord.

5.3. Other Test Personnel

Other test personnel may enter after clearance to collect samples, clean up the floor and remove any artifacts. The artifacts will be placed in a cleaned glass jar.

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1. BANGBOX LOW-DENSITY TRITONAL DETONATION TRIALS

1.1. Purpose

The purpose of these trials is to sample and analyze the emission products from the detonation of low-density Tritonal (80/20 - TNT/Al)

1.2. BangBox Preparation

The low-density charge will be suspended at least 1 m above the center of the cover over the firing pit.

2. TRITONAL DESIGNATION

2.1. Explosive

The explosive will be 227 g of low-density Tritonal enclosed in a thin polyethylene film.

2.2. Blasting Caps

Exploding bridge wire blasting caps are preferable but such systems may not be available for the test. Electric blasting caps will be used instead.

2.3. Explosive Preparation

2.3.1. Trinitrotoluene (TNT) Preparation. At a designated preparation site, three 454-g TNT demolition blocks (or 1.36 kg of flake TNT, Grade 1) will be lightly crushed with a wooden rolling pin on a cardboard. This will be sufficient to make six charges of Tritonal.

2.3.2. Tritonal Mixing. Place the weighed TNT from para. 2.3.1 in a glass jar. Add the exact weight of atomized aluminum, 340 g, to the TNT in the glass jar. Being sure there is no TNT on the rim, securely close the jar with a non-sparking lid. Gently roll and tumble the jar until the mixture is uniform.

2.3.3. Charge Preparation. Weigh out 227 g of the Tritonal mixture and place it in a polyethylene sandwich or freezer bag. As an alternate it may be placed in the center of a sheet of

thin polyethylene film and the edges gathered to form around the explosive. Secure the bag with a small cable tie or a piece of monofilament.

2.4. Support Material

2.4.1. Support Stands. Support stands will be placed opposite each other at the sides of the firing pit cover.

2.4.2. Charge Suspension. The charge will be suspended from the support stands with monofilament. The charge may be suspended vertically but the charge must be at least 1 m above the cover.

3. CHARGE PLACEMENT

3.1. Personnel

The charge will be brought to the Bang Box when ready for placement by qualified personnel only. All on site mounting will be under their direction and in accordance with established procedures.

3.2. Low-density Tritonal Charge Placement

3.2.1. Time of Placement. The charge will be brought into the BangBox and mounted after all of the other preparations and checking of test equipment are completed, and personnel not needed to set the charge are evacuated.

3.2.2. Procedure. The charge will be suspended from the support stands as shown in paragraph 2.4.2

3.2.3. Arming. The charge will be armed by inserting the cap in the top of the bag and fastening it with at least one cable tie. Monofilament line may alternately be used but no soft iron or other metal line will be used to mount or arm the charge.

4. INITIATION OF DETONATION.

4.1. Responsible Personnel

Initiation of detonation will be by qualified personnel only. They will be responsible for personnel control, functioning of interlocks, warning and initiation of the charge.

4.2. Firing Circuit Operation

Firing circuit operation will follow procedures prepared separately and appropriate to this and all other LOI.

5. BANGBOX REENTRY

5.1. Airlock

As required and approved limited test personnel may enter the air-lock adjacent to the BangBox to monitor instruments and obtain grab samples. They will not enter the BangBox itself until it has been cleared by the responsible weapons personnel.

5.2. Clearance

At the conclusion of the sampling period, responsible parties will enter the BangBox and inspect for any failures.

5.3. Other Test Personnel.

Other test personnel may enter after clearance to collect samples, clean up the floor and remove any artifacts. The artifacts will be placed in a cleaned glass jar.

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1. BANGBOX LOW-DENSITY TRITONAL WITH ADDITIVES DETONATION TRIALS

1.1. Purpose

The purpose of these trials is to sample and analyze the emission products from the detonation of low-density Tritonal (80/20 - TNT/Al) with additional organic additives to simulate non-explosive materials contained in bombs.

1.2. BangBox Preparation

The low-density Tritonal with additives charge will be suspended at least 1 m above the center of the cover over the firing pit.

2. TRITONAL WITH ADDITIVES DESIGNATION

2.1. Explosive

The explosive will be 220 g of low-density Tritonal, to which will be added 11 g of powdered organic additive. A suitable additive will be powdered calcium stearate, chosen because it is available as a fine powder and will simulate a wax.

2.2. Blasting caps

Exploding bridge wire blasting caps are preferable but such systems may not be available for the test. Electric blasting caps will be used instead.

2.3. Explosive Preparation

2.3.1. Tritonal Preparation. Low-density Tritonal prepared in LOI 31 will also be used in these trials.

2.3.2. Tritonal Mixing. Weigh Low-density Tritonal from that remaining from LOI 31 and place it in a clean glass jar. Weigh powdered calcium stearate equivalent to three percent and add to the Tritonal in the jar. Being sure that there is no explosive on the rim, close the bottle with a non-sparking lid and gently roll and tumble the jar until the mixture is uniform.

2.3.3. Charge Preparation. Weigh out 227 g of the Tritonal/additive mixture and place it in a polyethylene sandwich or freezer bag. As an alternate it may be placed in the center of a sheet of thin polyethylene film and the edges gathered to form around the explosive. Secure the bag with a small cable tie or a piece of monofilament.

2.4. Support Material

2.4.1. Support Stands. Support stands will be placed opposite each other at the sides of the firing pit cover.

2.4.2. Charge Suspension. The charge will be suspended from the support stands with monofilament. The charge may be suspended vertically but the charge must be at least 1 m above the cover.

3. CHARGE PLACEMENT

3.1. Personnel

The charge will be brought to the BangBox when ready for placement by qualified personnel only. All on site mounting will be under their direction and in accordance with established procedures.

3.2. Charge Placement

3.2.1. Time of Placement. The charge will be brought into the BangBox and mounted after all of the other preparations and checking of test equipment are completed, and personnel not needed to set the charge are evacuated.

3.2.2. Procedure

The charge will be suspended from the support stands as shown in paragraph 2.4.2

3.2.3. Arming. The charge will be armed by inserting the cap in the top of the bag and fastening it with at least one cable tie. Monofilament line may alternately be used but no soft iron or other metal line will be used to mount or arm the charge.

4. INITIATION OF DETONATION.

4.1. Responsible Personnel

Initiation of detonation will be by qualified personnel only. They will be responsible for personnel control, functioning of interlocks, warning and initiation of the charge.

4.2. Firing Circuit Operation

Firing circuit operation will follow procedures prepared separately and appropriate to this and all other LOI.

5. BANGBOX REENTRY

5.1. Airlock

As required and approved limited test personnel may enter the air-lock adjacent to the BangBox to monitor instruments and obtain grab samples. They will not be enter the BangBox itself until it has been cleared by the responsible weapons personnel.

5.2. Clearance

At the conclusion of the sampling period, responsible parties will enter the BangBox and inspect for any failures.

5.3. Other Test Personnel.

Other test personnel may enter after clearance to collect samples, clean up the floor and remove any artifacts. The artifacts will be placed in a cleaned glass jar.

INTENTIONALLY BLANK

1. BANGBOX LOW-DENSITY AMATOL DETONATION TRIALS

1.1. Purpose

The purpose of these trials is to sample and analyze the emission products from the detonation of low-density Amatol (50/50 - trinitrotoluene (TNT)/ammonium nitrate).

1.2. BangBox Preparation

The low-density Amatol charge will be suspended at least 1 m above the center of the cover over the firing pit.

2. AMATOL DESIGNATION

2.1. Explosive

The explosive will be 227 g of low-density Amatol.

2.2. Blasting Caps

Exploding bridge wire blasting caps are preferable but such systems may not be available for the test. Electric blasting caps will be used instead. M6 or larger blasting caps may be used.

2.3. Additional Primer

As an additional primer is necessary based on the trials of LOI 31, composition C-4 1.27 x 1.27 x 2.54 cm or about 6.5 g will be molded around the blasting cap before inserting into the charge.

2.4. Explosive Preparation

2.4.1. Amatol Preparation. Low-density Amatol will be prepared by crushing 454 g of TNT demolition block or 454 g of TNT flake, Grade 1. As approved by U.S. Army Dugway Proving Ground (DPG) and West Desert Test Center (WDTC) standing operating procedures (SOPs), a wooden rolling pin on paper or smooth plastic may gently crush the TNT. This TNT will then be mixed with an equal weight of fine-grained ammonium nitrate. Preferably the TNT will be crushed at an approved facility.

2.4.2. Amatol Mixing. Weigh the crushed TNT and place it in a clean glass jar. Weigh an equal amount of fine-grained ammonium nitrate and place it with the TNT in the jar. Be sure there is no explosive on the jar rim and close it with a non-sparking lid. Gently roll and tumble the jar until the mixture is uniform.

2.4.3. Charge Preparation. Weigh out 227 g of the low-density Amatol mixture and place it in a 1-qt (0.95L) polyethylene freezer bag. Secure the bag with a small tie or a piece of monofilament.

2.5. Support Material

2.5.1. Support Stands. Support stands will be placed opposite each other at the sides of the firing pit cover.

2.5.2. Charge Suspension. The charge will be suspended from the support stands with monofilament or cotton string. The charge may be suspended vertically but the charge must be at least 1 m above the cover.

3. CHARGE PLACEMENT

3.1. Personnel

The charge will be brought to the BangBox when ready for placement by qualified personnel only. All on-site mounting will be under their direction and in accordance with established DPG and WDTC safety procedures.

3.2. Charge Placement

3.2.1. Time of Placement. The charge will be brought into the BangBox and mounted after all of the other preparations and checking of test equipment are completed, and personnel not needed to set the charge are evacuated.

3.2.2. Procedure. The charge will be suspended from the support stands discussed above in paragraph 2.5.2

3.2.3. Arming. The charge will be armed by inserting the cap and any booster in the top of the bag and fastening it with at least one tie, string or monofilament line. but no soft iron or other metal wire will be used to mount or arm the charge except for the blasting cap leads.

4. INITIATION OF DETONATION.

4.1. Responsible Personnel

Initiation of detonation will be by qualified personnel only. They will be responsible for personnel control, functioning of interlocks, warning and initiation of the charge.

4.2. Firing Circuit Operation

Firing circuit operation will follow procedures prepared separately and appropriate to this and all other LOI.

5. BANGBOX REENTRY

5.1. Airlock

As required and approved limited test personnel may enter the air-lock adjacent to the BangBox to monitor instruments and obtain grab samples. They will not enter the BangBox itself until it has been cleared by the responsible and approved personnel.

5.2. Clearance

At the conclusion of the sampling period, responsible parties will enter the BangBox and inspect for any failures.

5.3. Other Test Personnel.

Other test personnel may enter after clearance to collect samples, clean up the floor and remove any artifacts. These artifacts will be placed in a cleaned glass jar.

6. REFERENCES

This LOI does not supersede DPG and WDTC SOP and regulations. Where there is a conflict, it will be resolved by the test officer before proceeding with the test.

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1. BANGBOX LOW-DENSITY RDX/TNT/WAX DETONATION TRIALS

1.1. Purpose

The purpose of these trials is to sample and analyze the emission products from the detonation of low-density composition B surrogate charge of RDX/trinitrotoluene (TNT)/wax (58/38/4).

1.2. BangBox Preparation

This low-density charge will be suspended at least 1 m above the center of the cover over the firing pit.

2. EXPLOSIVE DESIGNATION

2.1. Explosive

The explosive will be 227 g of low-density composition B surrogate charge.

2.2. Blasting Caps

Exploding bridge wire blasting caps are preferable but such systems may not be available for the test. Electric blasting caps will be used instead. M6 or larger blasting caps may be used.

2.3. Additional Primer

If an additional primer is necessary based on the previous trials of, composition C-4 1.27 by 1.27 by 2.54 cm or about 6.5 g will be molded around the blasting cap before inserting into the charge.

2.4. Explosive Preparation

2.4.1. Composition B Preparation

Low-density composition B will be prepared by crushing 908 g of TNT demolition block or 908 g of TNT flake, Grade 1. As approved by U.S. Army Dugway Proving Ground (DPG) and West Desert Test Center (WDTC) standing operating procedures (SOPs), a wooden rolling pin on paper or smooth plastic may gently crush the TNT. A weighed portion of TNT will then be

mixed with an appropriate weight of finely divided composition A-3 if it is available. The TNT will be crushed in an approved facility by approved personnel and approved procedure.

2.4.2. RDX component. Preferably, composition A-3 is desired to prepare this charge. If it is not on hand finely divided composition C-4 may be mixed well with the finely divided or crushed TNT.

2.4.3. Composition B Preparation. Weigh 348 g of crushed TNT and place it in a clean glass jar. Weigh 567 g of finely divided composition A-3 and place it in the glass jar. If composition C-4 is used, it must be broken or cut up in small pieces and the mixture with TNT carefully incorporated. Be sure there is no explosive on the jar rim and close it with a non-sparking lid. Gently roll and tumble the jar until the mixture is uniform.

2.4.4. Charge Preparation. Weigh out 227 g of the low-density composition B mixture and place it in a 1-pt (0.47-L) polyethylene freezer bag. Secure the bag with a small tie or a piece of monofilament. Mark the prepared charges and place them in paper bags for storage and transport.

2.4.5. Crushed TNT. Reserve the remainder of the crushed TNT for other charges.

2.5. Support Material

2.5.1. Support Stands. Support stands will be placed opposite each other at the sides of the firing pit cover.

2.5.2. Charge Suspension. The charge will be suspended from the support stands with monofilament or cotton string. The charge may be suspended vertically but the charge must be at least 1 m above the cover.

3. CHARGE PLACEMENT

3.1. Personnel

The charge will be brought to the BangBox when ready for placement by qualified personnel only. All on-site mounting will be under their direction and in accordance with established DPG safety procedures.

3.2. Charge Placement

3.2.1. Time of Placement. The charge will be brought into the BangBox and mounted after all of the other preparations and checking of test equipment are completed, and personnel not needed to set the charge are evacuated.

3.2.2. Procedure. The charge will be suspended from the support stands as discussed in paragraph 2.5.2

3.2.3. Arming. The charge will be armed by inserting the cap and any booster in the top of the bag and fastening it with at least one tie, string or monofilament line, but no soft iron or other metal wire will be used to mount or arm the charge except for the blasting cap leads.

4. INITIATION OF DETONATION.

4.1. Responsible Personnel

Initiation of detonation will be by qualified personnel only. They will be responsible for personnel control, functioning of interlocks, warning and initiation of the charge.

4.2. Firing Circuit Operation

Firing circuit operation will follow procedures prepared separately and appropriate to this and all other LOI.

5. BANGBOX REENTRY

5.1. Airlock

As required and approved limited test personnel may enter the air-lock adjacent to the BangBox to monitor instruments and obtain grab samples. They will not enter the BangBox itself until it has been cleared by the responsible and approved personnel.

5.2. Clearance

At the conclusion of the sampling period, responsible parties will enter the BangBox and inspect for any failures.

5.3. Other Test Personnel.

Other test personnel may enter after clearance to collect samples, clean up the floor and remove any artifacts. These artifacts will be placed in a cleaned glass jar for disposal by appropriate personnel.

6. REFERENCES

This LOI does not supersede DPG and WDTC SOP and regulations. Where there is a conflict, it will be resolved by the test officer before proceeding with the test.

This LOI includes the material safety data sheets (MSDS) on TNT and the other components that will be used in this charge. All precautions in these MSDS as applicable will be observed.

1. BANGBOX LOW-DENSITY RDX/TNT/AL/WAX DETONATION TRIALS

1.1. Purpose

The purpose of these trials is to sample and analyze the emission products from the detonation of low-density HBX and similar surrogate charge of RDX/trinitrotoleuen (TNT)/aluminum/wax (48.1/31.5/17/3.3).

1.2. BangBox Preparation

This low-density charge will be suspended at least 1 m above the center of the cover over the firing pit.

2. EXPLOSIVE DESIGNATION

2.1. Explosive

The explosive will be 227 g of low-density HBX-type surrogate charge.

2.2. Blasting Caps

Exploding bridge wire blasting caps are preferable but such systems may not be available for the test. Electric blasting caps will be used instead. M6 or larger blasting caps may be used.

2.3. Additional Primer

An additional primer as necessary, based on the previous trials, of composition C-4 1.27 by 1.27 by 2.54 cm or about 6.5 g will be molded around the blasting cap before inserting into the charge.

2.4. Explosive Preparation

2.4.1. HBX-Surrogate Charge Preparation. Low-density composition B surrogate mix prepared for LOI 34 will be the basis for this charge. Sufficient finely atomized aluminum powder will be mixed with it to make a charge with 17 percent aluminum.

2.4.2. RDX Component. Preferably, composition A-3 is desired to prepare this charge. Since it was not on hand finely divided composition C-4 will be well mixed with crushed TNT as described in LOI 35, Rev 1.

2.4.3. HBX-Surrogate Base Charge Preparation. Weigh 348 g of crushed TNT and place it in a clean glass jar. Weigh 567 g of composition C-4 and form it into very small pieces and tumble the jar after a few small pieces of C-4 are added to keep it coated with TNT. When all of the composition C-4 has been added the glass jar may be closed up, being sure that no explosive is present on the threads or the mouth of the jar. Rotate, tumble and work the mixture until it is uniform.

2.4.4. Aluminum Addition. When the base charge in 2.4.3 is complete, weigh out 187 g of finely atomized aluminum. Pour this aluminum into the base charge in the bottle. Be sure that there is no material on the threads or rim of the bottle and close it securely. Rotate and tumble the bottle until the charge is uniformly mixed.

2.4.5. Test-Charge Preparation. Weigh out 227 g of this low-density HBX-surrogate mixture and place it in a 1-pt (0.47-L) polyethylene freezer bag. Secure the bag with a small tie or a piece of monofilament. Prepare three charges of this mixture. Reserve the remainder of the explosive for future use. Mark the prepared charges and place them in paper bags for storage and transport.

2.5. Support Material

2.5.1. Support Stands. Support stands will be placed opposite each other at the sides of the firing pit cover.

2.5.2. Charge Suspension. The charge will be suspended from the support stands with monofilament or cotton string. The charge may be suspended vertically but the charge must be at least 1 m above the cover.

3. CHARGE PLACEMENT

3.1. Personnel

The charge will be brought to the BangBox when ready for placement by qualified personnel only. All on-site mounting will be under their direction and in accordance with established U.S. Army Dugway Proving Ground (DPG) and West Desert Test Center (WDTC) safety procedures.

3.2. Charge Placement

3.2.1. Time of Placement. The charge will be brought into the BangBox and mounted after all of the other preparations and checking of test equipment are completed, and personnel not needed to set the charge are evacuated.

3.2.2. Procedure. The charge will be suspended from the support stands as discussed in paragraph 2.5.2

3.2.3. Arming. The charge will be armed by inserting the cap and any booster in the top of the bag and fastening it with at least one tie, string or monofilament line. but no soft iron or other metal wire will be used to mount or arm the charge except for the blasting cap leads.

4. INITIATION OF DETONATION.

4.1. Responsible Personnel

Initiation of detonation will be by qualified personnel only. They will be responsible for personnel control, functioning of interlocks, warning and initiation of the charge.

4.2. Firing Circuit Operation

Firing circuit operation will follow procedures prepared separately and appropriate to this and all other LOI.

5. BANGBOX REENTRY

5.1. Airlock

As required and approved limited test personnel may enter the air-lock adjacent to the BangBox to monitor instruments and obtain grab samples. They will not enter the BangBox itself until it has been cleared by the responsible and approved personnel.

5.2. Clearance

At the conclusion of the sampling period, responsible parties will enter the BangBox and inspect for any failures.

5.3. Other Test Personnel.

Other test personnel may enter after clearance to collect samples, clean up the floor and remove any artifacts. These artifacts will be placed in a cleaned glass jar.

6. REFERENCES

This LOI does not supersede DPG and WDTC standing operating procedures and regulations. Where there is a conflict, it will be resolved by the test officer before proceeding with the test.

This LOI includes the material safety data sheets on TNT, composition C-4 and aluminum which will be used in this charge. All applicable precautions will be observed.

1. BANGBOX LOW-DENSITY TRITONAL WITH WATER DETONATION TRIALS

1.1. Purpose

The purpose of these trials is to sample and analyze the emission products from the detonation of low-density Tritonal (80/20 - trinitrotoluene (TNT)/aluminum) when detonated surrounded by water. As part of this purpose, the effect of water on fireball quenching will be observed.

1.2. BangBox Preparation

The low-density charge surrounded by water will be suspended at least 1 m above the center of the cover over the firing pit.

2. TRITONAL DESIGNATION

2.1. Explosive

The explosive will be 227 g of low-density Tritonal enclosed in a thin polyethylene film.

2.2. Blasting Caps

Exploding bridge wire blasting caps are preferable but such systems may not be available for the test. Electric blasting caps will be used instead.

2.3. Explosive Preparation

2.3.1. Tritonal Preparation. Explosives technicians will crush TNT at the designated preparation site and prepare Tritonal charges to study low-density Tritonal as described in letter or instruction (LOI) 31 of this study. Three charges will be weighed and designated as F-1, F-2 and F-3 each weighing 227 g.

2.3.2. Additional Charges. If additional charges are needed, they may be mixed from available fine TNT and available aluminum powder.

2.3.3. Charge Preparation. The test charges for this study will be packed in 1-pt (0.47-L) capacity thin polyethylene bags. When ready, the this charge will be placed in a 1-qt (0.95-L) polyethylene bag before immersing it in water. This will prevent water from entering the charge as it is immersed.

2.3.4. Water Surround. The charge will be surrounded by four to six times its weight in water. This water will be contained in a transparent container allowing water to uniformly surround the charge. One-quart (0.95-L) bags with water will surround the charge and be held in place by a larger bag.

2.4. Support Material

2.4.1. Support Stands. Support stands will be placed opposite each other at the sides of the firing pit cover.

2.4.2. Charge Suspension. The charge will be suspended from the support stands with monofilament line or cotton string. The water container will then be suspended independently from the string.

3. CHARGE PLACEMENT

3.1. Personnel

The charge will be brought to the BangBox when ready for placement by qualified personnel only. All on site mounting will be under their direction and in accordance with established procedures.

3.2. Low-density Tritonal Charge with Water Placement

3.2.1. Time of Placement. The charge will be brought into the BangBox and mounted after all of the other preparations and checking of test equipment are completed, and personnel not needed to set the charge are evacuated.

3.2.2. Procedure. The charge will be suspended from the support stands as described in paragraph 2.4.2.

3.2.3. Arming. The charge will be armed by inserting the cap in the top of the bag and fastening it with at least one cable tie. Monofilament line or cotton string may alternately be used but no soft iron or other metal line will be used to mount or arm the charge.

4. INITIATION OF DETONATION.

4.1. Responsible Personnel

Initiation of detonation will be by qualified personnel only. They will be responsible for personnel control, functioning of interlocks, warning and initiation of the charge.

4.2. Firing Circuit Operation

Firing circuit operation will follow procedures prepared separately and appropriate to this and all other LOI.

5. BANGBOX REENTRY

5.1. Air Lock

As required and approved limited test personnel may enter the air-lock adjacent to the BangBox to monitor instruments and obtain grab samples. They will not enter the BangBox itself until it has been cleared by the responsible weapons personnel.

5.2. Clearance

At the conclusion of the sampling period, responsible parties will enter the BangBox and inspect for any failures.

5.3. Other test personnel.

Other test personnel may enter after clearance to collect samples, clean up the floor and remove any artifacts. The artifacts will be placed in a cleaned glass jar.

6. REFERENCES

6.1. This LOI does not supersede U.S. Army Dugway Proving Ground or West Desert Test Center standing operating procedures and regulations. Where there is a conflict, it will be resolved by the Test Officer before proceeding with the test.

6.2. This LOI includes the material safety data sheets on TNT and aluminum which will be used in this charge. All applicable precautions will be observed.

1. BANGBOX LOW-DENSITY AMATOL WITH WATER DETONATION TRIALS

1.1. Purpose

The purpose of these trials is to sample and analyze the emission products from the detonation of low-density Amatol (50/50 - trinitrotoluene (TNT)/ammonium nitrate) when detonated surrounded by water. As part of this purpose, the effect of water on fire ball quenching will be observed.

1.2. BangBox Preparation

The low-density charge surrounded by water will be suspended at least 1 m above the center of the cover over the firing pit.

2. AMATOL DESIGNATION

2.1. Explosive

The explosive will be 227 g of low-density Amatol enclosed in a thin polyethylene film.

2.2. Blasting Caps

Exploding bridge wire blasting caps are preferable but such systems may not be available for the test. Electric blasting caps will be used instead.

2.3. Explosive Preparation

2.3.1. Amatol Preparation. At the approved facility TNT was crushed and Amatol charges prepared to study low density Amatol as described in LOI 33 of this study. This TNT will be mixed with an equal weight of fine grained ammonium nitrate.

2.3.2. Additional Charges. No additional charges were prepared in the original composition. For this LOI they may be mixed from available fine TNT and available ammonium nitrate using the procedure of LOI 33, paragraph 2.4.

2.3.3. Charge Preparation. The test charges for this study will be packed in 1-pt (0.47-L) capacity thin polyethylene bags. When ready, this charge will be placed in a 1-gal (3.79-L) freezer bag after priming to prevent water from entering the charge.

2.3.4. Water-Surround Preparation. The charge will be surrounded by four to six times its weight in water. The charge when primed will be placed in a 1-gal (3.79-L) freezer bag and the bag pulled tight around the charge. Five 1-qt (0.95-L) freezer bags will each receive 1 1/3 cups of water and the top securely fastened near the top of the bag. One of these bags will be placed in the bottom of a 1-gal (3.79-L) freezer bag. Three of the bags will be placed vertically inside of the bag and the charge will be placed between them. The fifth bag will be placed on top of the charge.

2.4. Support Material

2.4.1. Support Stands

Support stands will be placed opposite each other at the sides of the firing pit cover.

2.4.2. Charge Suspension

The charge will be suspended from the support stands with at least a double strand of string. The three water bags will then be suspended from the string. The gallon bag with the quart bag in the bottom will be raised up around the charge, the upper placed in it and the top of the gallon bag secured.

3. CHARGE PLACEMENT

3.1. Personnel

The charge will be brought to the BangBox when ready for placement by qualified personnel only. All on site mounting will be under their direction and in accordance with established procedures.

3.2. Low-Density Amatol Charge with Water Placement

3.2.1. Time of Placement. The charge will be brought into the BangBox and mounted after all of the other preparations and checking of test equipment are completed, and personnel not needed to set the charge are evacuated.

3.2.2. Procedure. The charge will be suspended from the support stands as described in paragraph 2.4.2

3.2.3. Arming. The charge will be armed by inserting the cap in the top of the bag and fastening it with at least one cable tie. Monofilament line or cotton string may alternately be used but no soft iron or other metal line will be used to mount or arm the charge.

4. INITIATION OF DETONATION

4.1. Responsible Personnel

Initiation of detonation will be by qualified personnel only. They will be responsible for personnel control, functioning of interlocks, warning and initiation of the charge.

4.2. Firing Circuit Operation

Firing circuit operation will follow procedures prepared separately and appropriate to this and all other LOI.

5. BANGBOX REENTRY

5.1. Airlock

As required and approved limited test personnel may enter the air-lock adjacent to the BangBox to monitor instruments and obtain grab samples. They will not enter the BangBox itself until it has been cleared by the responsible weapons personnel.

5.2. Clearance

At the conclusion of the sampling period, responsible parties will enter the BangBox and inspect for any failures.

5.3. Other Test Personnel

Other test personnel may enter after clearance to collect samples, clean up the floor and remove any artifacts. The artifacts will be placed in a cleaned glass jar.

West Desert Test Center
Complex Munitions Test
LOI 37, Rev 1
Prepared by ECO, L.C.
23 August 1995
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6. REFERENCES

This LOI does not supersede U.S. Army Dugway Proving Ground and West Desert Test Center standing operating procedures and regulations. Where there is a conflict, it will be resolved by the test officer before proceeding with the test.

This LOI includes the material safety data sheets on TNT and ammonium nitrate will be used in this charge. All applicable precautions will be observed.

1. ALUMINIZED COMPOSITE PROPELLANT WITH WASTE BURN TESTS

1.1. Propellant/Waste Composition

The propellant/waste composition will be 65 percent aluminized propellant, 20 percent polyethylene, 11 percent paper and cloth, 4 percent diesel fuel. The propellant will be furnished in pieces to simulate actual contaminated waste.

1.2. Charge Preparation

1.2.1. Personnel. Aluminized composite propellant will only be handled by qualified personnel. Impervious gloves will be worn during charge preparation and at any time the bare propellant and waste are handled. Only necessary personnel will be present during charge preparation.

1.2.2. Location. Aluminized propellant/waste charges will be prepared at a site remote from the BangBox.

1.2.3. Size of Charge. The initial charge size will contain 1,135 g of propellant. After the first sample burn, adjustment of charge will be made as needed to control sampler particulate and chlorine compound loadings. The propellant for this charge will be weighed and stored in a separate paper bag for each of the three replicate burns. It will be transported to the BangBox in this container.

1.2.4 Ignition Charge Requirement. Four grams of Hercules Unique™ smokeless powder in a small cloth bag will be used to assure reliable ignition. This ignition charge will be prepared away from the BangBox.

1.2.4. Storage. Each propellant charge will be stored in a paper wrapping, bag or tube after preparation for storage and subsequent transport to the BangBox.

2. TEST PROCEDURES

2.1. Charge Location

The charge will be placed at the site in the BangBox prepared by project personnel in accordance with ECO LOI 20.

2.2. Charge Placement Procedures

2.2.1. Inspection. The site will be inspected before charge placement to assure adequate cleanliness and that the firing cable is properly shunted.

2.2.2. Charge Assembly. The aluminized composite pieces will be placed in the center of the stainless steel burn pan and intermixed with the non-reactive waste in a manner which is representative of actual waste products. An amount of diesel fuel equivalent to the 4 percent proportion in the waste will be added.

2.2.3. Igniter Placement. The bag containing the Hercules Unique™ smokeless powder will be placed between two propellant pieces away from diesel fuel.

2.3. Charge Arming

2.3.1. Electric Squib Handling. Each electric squib will be tested with a galvanometer and two squibs selected. These squibs will then be connected to the firing line and any shunts on the squibs themselves will be removed.

2.3.2. Squib Insertion. The two squibs will be inserted into the top of the Hercules Unique™ smokeless powder bag to be in contact with the Hercules Unique™ smokeless powder material.

2.3.3. Removal of Packaging. After insertion of the squibs, personnel will remove all packing materials for charge transport for proper disposal.

3. CHARGE-IGNITION PROCEDURES

3.1. Evacuation

Ignition personnel will not leave the BangBox airlock until all other personnel have departed for the command post (CP). Positive pressure in the airlock will be maintained by keeping the blower on during actual sampling procedures.

3.2. Firing Line Inspection

Ignition personnel will remove the shunt in the exterior box after leaving the BangBox airlock. They will then proceed to the CP.

3.3. Command Post Procedures

3.3.1. Personnel. Before proceeding with charge ignition, the charge-ignition personnel will assure themselves that all personnel are accounted for. It will be the responsibility of the Test Officer or his designee to control all observers.

3.3.2. Readiness. Before proceeding with the ignition, the charge-ignition personnel will obtain an ignition time from the technicians responsible for data acquisition. These technicians are responsible to assure that all systems are ready to acquire test data.

3.3.3. Charge Ignition

The charge-ignition personnel will prepare the box for firing and with an appropriate count down ignite the charge. Ignition will be confirmed by video observation or by the data being collected.

4. POST-IGNITION PROCEDURES

4.1. Sampling Personnel

Charge-ignition personnel will clear only essential sample gathering personnel to proceed to the BangBox airlock to collect gas samples. These personnel are not cleared to enter the BangBox. They will be sure the positive pressure blower is operating before beginning sampling and will have respirators available which will be suitable for high particulate and HCl or Cl₂ loadings are found in the air lock.

4.2. Post-Sampling Inspection

4.2.1. Air Sampling. After the sampling period is complete, the charge sampling personnel will inspect the BangBox interior under mask and open the vent door. They will sampling the air with a Drager unit and will signal the firing personnel when it is safe to enter.

4.2.2. Clearing the BangBox for Propellant Safety. Firing personnel will gather any unburned propellant in appropriate containers. They will then clear sampling personnel to enter the BangBox to collect samples and prepare for the next burn.

4.3. Personnel Access

Further post burn access after to the BangBox will be as designated by the Test Officer.

5. ACCOUNTABILITY

5.1. Propellant Loading

5.1.1. Records. Records will be kept of the weight of propellant, descriptive numbers and condition and other pertinent information.

5.1.2. Compliance to LOI. Records will be kept of loading conditions. Loading sheets will be completed describing all charge conditions and procedures according to this LOI.

6. REFERENCES

6.1. West Desert Test Center LOI 20: "Propellant Burn Test - BangBox."

6.2. U.S. Army Dugway Proving Ground and West Desert Test Center standing operating procedures prepared for BangBox testing.

6.3. *Draft TEST DESIGN PLAN for Characterization of Mixing Profile and HCl/Cl₂ Recovery of Emissions Produced During Thermal Treatment of Propellants*, July 1995.

1. DIESEL FUEL/DUNNAGE WASTE BURN TESTS

1.1. Diesel Fuel/Dunnage Waste Composition

The diesel-fuel/dunnage waste composition will be nominally 60 percent penta-treated wood, 15 percent pasteboard of various types, 12.5 percent kraft paper, 2.5 percent Styrofoam to which will be added 10 percent diesel fuel. This is intended to somewhat simulate an actual bonfire used in the field to destroy a variety of small munitions.

1.2. Charge Preparation

1.2.1. Personnel. Diesel-fuel/dunnage charges will be assembled by competent and approved personnel.

1.2.2. Location. Diesel-fuel/dunnage charges will be prepared at a site remote from the BangBox.

1.2.3. Size of Charge. The initial charge will be approximately 908 g total weight.

1.2.4. Component Preparation

1.2.4.1. Wood. The wood used in these trials will be from an excess ammunition storage box which is expected to have been treated with pentachlorophenol as a preservative. This wood will be cut in 0.64 to 1.27-cm thick strips 25.5 to 30.5 cm in length. Each individual charge will contain 545 g of this wood.

1.2.4.2. Pasteboard. The pasteboard for these trials will be a mixture of corrugated and plain cardboard such as may be expected from excess dunnage. A total of 136 g of mixed pieces will be weighed for an individual charge.

1.2.4.3. Paper. Various types of kraft paper will be used in these trials. Each trial will be composed 114 g of the same mixture of the various papers available.

1.2.4.4. Styrofoam. Rigid Styrofoam will be used. Each trial will use 23 g of this product.

1.2.4.5. Diesel Fuel. Ninety-one grams of the diesel fuel obtained for previous trials of this test series will be weighed out for each trial. The diesel fuel will be in a separate glass jar.

1.2.5. Storage and Transport. The individual trials will be weighed and stored in a separate paper bag for each of the three replicate burns. It will be transported to the BangBox in this container. This paper bag will be included as a part of the paper component of the trial.

1.2.6. Ignition-Charge Requirement. An ignition supplement in a small cloth bag to ensure reliable ignition will contain 4 g of Hercules Unique™ smokeless powder. This ignition charge will be prepared away from the BangBox.

1.2.7. Storage. Except for the ignition charge, no special storage is required for these trials.

2. TEST PROCEDURES

2.1. Charge location

The charge will be placed at the site in the BangBox prepared by project personnel in accordance with ECO LOI 20.

2.2. Charge Placement Procedures

2.2.1. Inspection. The site will be inspected before charge placement to assure adequate cleanliness and that the firing cable is properly shunted.

2.2.2. Charge Assembly. The individual components may be placed in the same large kraft paper bag except for the diesel oil. In the BangBox the paper and pasteboard will be placed and concentrated in the bottom of a burn pan. The Styrofoam and the wood pieces will be stacked over the wood somewhat in tepee fashion. When ready to ignite the charge, the diesel fuel will be poured over the entire charge.

2.2.3. Igniter Placement. The bag containing the Hercules Unique™ smokeless powder will be placed near the bottom of the assembly in contact with the paper

2.3. Charge Arming

2.3.1. Electric Squib Handling. Only reliable electric squibs will be used in these trials. These squibs will be connected to the firing line and any shunts on the squibs themselves will be removed.

2.3.2. Squib Insertion. The two squibs will be inserted into the top of the Hercules Unique™ smokeless powder bag in direct contact with the powder.

2.3.3. Removal of Packaging. After insertion of the squibs, personnel will remove excess packing materials for charge transport for proper disposal.

3. CHARGE-IGNITION PROCEDURES

3.1. Evacuation

Ignition personnel will not leave the BangBox airlock until all other personnel have departed for the command post (CP). Positive pressure in the airlock will be maintained by keeping the blower on during actual sampling procedures.

3.2. Firing Line Inspection

Ignition personnel will remove the shunt in the exterior box after leaving the BangBox airlock. They will then proceed to the CP.

3.3. Command Post

3.3.1. Personnel. Before proceeding with charge-ignition, the charge-ignition personnel will assure themselves that all personnel are accounted for. It will be the responsibility of the test officer or his designee to control all observers.

3.3.2. Readiness

Before proceeding with the ignition, the charge-ignition personnel will obtain an ignition time from the technicians responsible for data acquisition. These technicians are responsible to assure that all systems are ready to acquire test data.

3.3.3 Charge Ignition. The charge-ignition personnel will prepare the box or firing and with an appropriate count down ignite the charge. Ignition will be confirmed by video observation or by the data being collected.

4. POST-IGNITION PROCEDURES

4.1. Sampling Personnel

Charge-ignition personnel will clear only essential sample gathering personnel to proceed to the BangBox airlock to collect gas samples. These personnel are not cleared to enter the BangBox. They will be alert and prepared for high particulate and HCl or Cl₂ loadings are found in the air lock.

4.2. Post-Sampling inspection

4.2.1. Air Sampling. After the sampling period is complete, the charge sampling personnel will inspect the BangBox interior under mask and open the vent door. They will sampling the air with a Drager unit if necessary and will signal the firing personnel when it is safe to enter.

4.2.2. Clearing the BangBox for Propellant Safety. Firing personnel will gather any unburned charge in appropriate containers. They will then clear sampling personnel to enter the BangBox to collect samples and prepare for the next burn.

4.3. Personnel Access

Further post burn access after to the BangBox will be as designated by the test officer.

5. ACCOUNTABILITY

5.1. Charge loading

5.1.1. Records. Records will be kept of the weight of components, descriptive numbers if any and conditions and other pertinent information.

5.1.2. Compliance to LOI. Records will be kept of loading conditions. Loading sheets will be completed describing all charge conditions and procedures according to this LOI.

6. REFERENCES

6.1. West Desert Test Center LOI 20: "Propellant Burn Test - BangBox"

6.2. U.S. Army Dugway Proving Ground and West Desert Test Center standing operating procedures prepared for BangBox testing.

6.3. Draft *TEST DESIGN PLAN for Characterization of Mixing Profile and HCl/Cl₂ Recovery of Emissions Produced During Thermal Treatment of Propellants*, July 1995.

6.4. Material safety data sheets if any are applicable.

6.5. T.O. 11A-1-42 General Instructions for Disposal of Conventional Munitions, 1 March 1979, Change 21 - 30 May 1991.

6.6. NIOSH Pocket Guide to Chemical Hazards, June 1990, Page 174; US Dept of Health and Human Services, U.S.G.P.O.

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APPENDIX I. LETTERS OF INSTRUCTION

MOUNTAIN STATES ANALYTICAL, INC

Title	LOI
Handling of Data	001
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SUBCONTRACT LABORATORY DOCUMENTS

Sunset Laboratories: Thermal-Optical Analysis for Organic/Elemental Carbon Aerosol	SNST001
MHW Laboratories: Standard Procedure for the Micro-Determination of Carbon and Hydrogen	MHW001
MHW Laboratories: Standard Modified Dumas Procedure for the Micro- Determination of Nitrogen	MHW002
MHW Laboratories: Cahn Balance Calibration Procedure	MHW003

Handling of Data

Recording and Correction of Data

1. All data are recorded in black permanent ink.
2. All dates are written in the order of day, month, year, the month being a three-letter abbreviation (*i.e.*, 25 Nov 1988).
3. All times are stated according to a twenty-four hour clock with a colon separating the hour from the minute (*i.e.*, 13:52).
4. All data sheets are signed and dated when completed and only after an inspection for completeness and correctness.
5. All notebooks are dated at the beginning of each day's entries and signed at the end of each day's entries and at least once on each page.
6. Incorrect data is crossed out with a single line such that it remains legible and the correct data is written in with the date of correction and the initials of the person making the correction.
7. Any reprocessing is considered new data and not a correction, and is handled as such.

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Preparation, Handling, and Storage of Quartz Fiber Filters and Filter Samples

A. Handling of Quartz Fiber Filters and Filter Samples

1. Clean cotton (100%) or nylon gloves are worn whenever handling filters or filter samples to maintain sample integrity. Disposable latex or nitrile gloves may be worn under cotton or nylon gloves to protect hands.
2. Filter samples and clean filters are allowed to contact only clean cotton or nylon gloves or clean, dry surfaces.

B. Preparation of Filters

1. Approximately 40 quartz fiber filters (8 in. x 10.5 in.) are rolled into a cylinder about 3.7 in. in diameter and placed into a 1,700 mL Soxhlet extraction tube.
2. The filters are extracted with 2,300 mL ACN as per LOI-009 for 24 h or more at a solvent temperature of 80 to 81°C and a reflux rate of approximately 3 per h.
3. The filters are dried for 1 h at 180 to 200°C.
4. The filters are allowed to equilibrate with the moisture in the air for at least 24 h.
5. The filters are inspected for pinholes or other flaws that might adversely affect performance.

C. Weighing of Filters and Filter Samples

1. The filters are weighed on a Mettler Toledo AB104 analytical balance with a filter weighing chamber to 0.1 mg both before and after sampling.
2. Prior to sampling, each filter is stored in a clean wide mouth bottle of at least 8 in. internal height and closed with a Teflon lined cap. The bottle

is labeled with sample number, filter number, filter weight, and the date that the filter was weighed.

3. After sampling and prior to weighing, filter samples are folded in fourths, returned to their bottles, and stored in dry ice or at $4 \pm 2^{\circ}\text{C}$ or $-15 \pm 5^{\circ}\text{C}$.
4. Filter samples are allowed to warm to room temperature for 30 min. prior to weighing.
5. After weighing, 15 circular sections (1.78 in. diameter) are cut from each designated filter for total carbon and/or metals analyses. One section for carbon analysis is suspended inside of a polypropylene specimen container. The remaining circular sections are placed together in pairs (sample sides in) and placed in another polypropylene specimen container. The 14 circular sections are for the following purposes: 4 sections for ICP analysis, 2 for CVAA analysis, 4 for quality control and/or archive, and 4 for archive. In addition, 10 circular sections (1.13 in. diameter) are cut from each designated filter for anion analysis. The sections are paired (sample sides in) and placed in a specimen container. Each specimen container is labeled with the identity of the contents. The filter remaining after removal of the circular sections is returned to its bottle. All containers holding filter sample media are stored at $4 \pm 2^{\circ}\text{C}$ or $-15 \pm 5^{\circ}\text{C}$ prior to analysis.
6. Filter samples designated for SFC/MS and SFC/TEA or GC/MS analysis are folded to retain particulate matter and placed in Soxhlet tubes for extraction or replaced in the bottles and stored at $4 \pm 2^{\circ}\text{C}$ or $-15 \pm 5^{\circ}\text{C}$ prior to extraction.
7. To ensure the accurate performance of the balance during weighing, the zero is checked before every weighing and adjusted as necessary. A standard reference weight in the same weight range as the samples is weighed at a frequency of once in every ten samples or less if fewer than ten samples are weighed. The standard reference weight set is checked monthly against a set of weights traceable to NIST Class S standards.
8. Weights are recorded in the laboratory notebook.

D. Storage of Filter Samples and Clean Filters

1. Filter samples and clean filters are stored in glass bottles with Teflon lined caps. The bottles are cleaned to U.S. EPA Contract Laboratory Program specifications. Bottles that have held samples are not cleaned and reused, but are discarded to prevent sample cross contamination.
2. Filter samples are stored at $4 \pm 2^{\circ}\text{C}$ or $-15 \pm 5^{\circ}\text{C}$.

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Cleaning, Handling, Storage, and Use of Burn Pans, Wire Brushes, Tote Boxes, and Filter Screens

A. Cleaning, Handling, and Storage of Burn Pans and Wire Brushes

1. Nitrile (NBR) gloves formulated without plasticizers and inert fillers are worn when cleaning new burn pans and wire brushes.
2. Pans are washed in soap and hot water, rinsed with deionized water and HPLC grade acetone, and dried in air for 12 h or more.
3. Brushes are washed in soap and hot water, rinsed with deionized water and HPLC grade acetone, and heated for 1 h or more at 90 to 100°C.
4. Clean cotton (100%) or nylon gloves are worn when handling clean burn pans or used burn pans prior to disposal or clean brushes or brushes in use to maintain sample integrity. This includes loading clean and used pans with test material and collecting residue from pans. Disposable latex or nitrile gloves may be worn under cotton or nylon gloves to protect hands.
5. Pans and brushes are allowed to cool on a clean, dry surface prior to storage in tote boxes. Used pans and brushes are not stored in the tote boxes.
6. Used burn pans are not cleaned but are discarded to prevent cross contamination of samples.
7. Used wire brushes are cleaned and reused.

B. Cleaning of Tote Boxes

1. Nitrile (NBR) gloves are worn when cleaning tote boxes.

2. The boxes are washed in soap and hot water, rinsed with deionized water and HPLC grade acetone or methanol, and air dried for 12 h prior to use.

C. Use of Burn Pans and Wire Brushes

1. A clean burn pan is used for the first combustion test of the day and/or whenever the test material is changed.
2. A clean wire brush is used to remove the residue from the pan following the first combustion test of the day and/or whenever the test material is changed.
3. The same pan and brush may be used for subsequent tests of the same test material on the same day. Residue should be removed from the pan following each test.
4. When setting down a brush that is still in use, care should be taken to ensure that the bristles not be contaminated.

D. Cleaning and Handling of Filter Screens

1. Nitrile (NBR) gloves are worn when cleaning filter screens.
2. Filter screens are scrubbed in an acetone/deionized water (50/50) solution, rinsed in an acetone/deionized water (50/50) solution, rinsed in deionized water, and heated for 6 h or more at 50 to 55°C.
3. Clean cotton or nylon gloves are worn when handling clean filter screens or screens in use to maintain sample integrity.
4. Filter screens are allowed to cool prior to storage in tote boxes. Used filter screens are not stored in the tote boxes.

Mountain States Analytical, Inc.

LOI-003, Rev. 1

22 February 1995

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Collection, Handling, and Storage of Burn Pan Residues

A. Handling of Burn Pan Residues

1. Clean cotton (100%) or nylon gloves are worn when handling burn pan residues in open containers to maintain sample integrity. Disposable latex or nitrile gloves may be worn under cotton or nylon gloves to protect hands.
2. Direct contact of the residues with hands is avoided.
3. Residues are allowed to contact only clean, dry surfaces.

B. Collection of Residues

The residue remaining after the combustion of a test material is removed from the burn pan with a wire brush (LOI 003), placed in a bottle cleaned to U.S. EPA Contract Laboratory specifications, and stored in dry ice or at $4 \pm 2^\circ\text{C}$ or $-15 \pm 5^\circ\text{C}$. Bottles that have held samples are not cleaned and reused.

C. Weighing of Residues

1. Residues are weighed to four significant digits or to 0.1 mg on an analytical balance.
2. Residues are allowed to warm to room temperature for 30 min. prior to weighing.

D. Storage of Residues

1. Residues are stored in bottles cleaned to U.S. EPA Contract Laboratory Program specifications.

2. Sample bottles containing residues are stored in dry ice or at $4 \pm 2^{\circ}\text{C}$ or $-15 \pm 5^{\circ}\text{C}$.

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Extraction of Quartz Fiber Filter Samples for SFC/MS and SFC/TEA and Handling of Extracts

A. Extraction of Quartz Fiber Filter Samples

1. Each filter sample is carefully folded to prevent particulate loss, spiked with 2 μg of 9-phenyl anthracene (internal standard), and extracted with 700 mL of HPLC grade (or better) acetonitrile (ACN) in a 500 mL Soxhlet extractor as per LOI-009 for 6 h at a solvent temperature of 80 to 81°C.
2. For every extraction event, a method blank consisting of 3 clean filters is extracted.
3. For every 20 samples extracted, a laboratory control sample (LCS) and an LCS duplicate (LCSD) are prepared and extracted. An LCS or an LCSD consists of 3 blank filters spiked with a solution containing 4 μg each of the target analytes listed in Table 5.1.

B. Handling of Extracts

1. The extract is concentrated to approximately 1 to 2 mL volume using a rotary evaporator (<28°C) as per LOI-010.
2. Each sample extract is split into 2 aliquots.
3. One aliquot is archived in an amber vial with a Teflon lined cap.
4. The other aliquot is eluted with ACN through solid phase extraction tubes (Alltech Sample Preparation Silica, 30 to 70 μm , 60Å) until a total volume of 4 mL is collected in an amber vial, concentrated to 0.2 mL under N_2 purge, and spiked with 1 μg of 1-nitronaphthalene- d_7 (internal standard) for SFC/MS and SFC/TEA analyses.

C. Storage of Extracts

Vials containing the filter extracts are stored at $4 \pm 2^\circ\text{C}$ or $-15 \pm 5^\circ\text{C}$.

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Table 5.1. Target Analytes in the LCS

1.	Nitroglycerine
2.	2,6-Dinitrotoluene
3.	2,4-Dinitrotoluene
4.	Pentaerythritol tetranitrate
5.	2-Nitronaphthalene
6.	2,4,6-Trinitrotoluene
7.	2-Nitrodiphenylamine
8.	1,3,5-Trinitrobenzene
9.	N-Methyl-N,2,4,6_tetranitroaniline
10.	Hexahydro-1,3,5-trinitro-1,3,5-triazine
11.	1-Nitropyrene
12.	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
13.	Naphthalene
14.	Phenol
15.	Dibenzofuran
16.	N-Nitrosodiphenylamine

Table 5.1. (Continued)

17.	Diphenylamine
18.	Pyrene
19.	Benz[α]anthracene
20.	Benzo[α]pyrene

Extraction of Quartz Fiber Filter Samples for GC/MS and Handling of Extracts

A. Extraction of Quartz Fiber Filter Samples

1. Each filter sample is carefully folded to prevent particulate loss, spiked with 8 μg of base and neutral surrogates (nitrobenzene- d_5 , 2-fluorobiphenyl, and terphenyl- d_{14}) and 16 μg of acid surrogates (2-fluorophenol, phenol- d_6 , and 2,4,6-tribromophenol), and extracted with 700 mL of HPLC grade (or better) methylene chloride (MeCl_2) in a 500 mL Soxhlet extractor as per LOI-009 for 6 h at a solvent temperature of 39 to 40°C.
2. For every extraction event, a method blank consisting of 3 clean filters is extracted.
3. For every 20 samples extracted, a laboratory control sample (LCS) and an LCS duplicate (LCSD) are prepared and extracted. An LCS or an LCSD consists of 3 blank filters spiked with a solution containing 20 μg each of target analytes listed in U.S. EPA SW-846 Method 8270A.

B. Handling of Extracts

1. The extract is dried by passing it through approximately 7g of anhydrous sodium sulfate.
2. The extract is concentrated in a Kuderna-Danish apparatus to approximately 1 to 2 mL volume (70 to 80°C) as per U.S. EPA SW-846 Method 3500.
3. Each sample extract is split into 2 aliquots.
4. One aliquot is archived in an amber vial with a Teflon lined cap.
5. The other aliquot is concentrated to 0.2 mL under N_2 purge (21 to 24°C).

C. Storage of Extracts

Vials containing the filter extracts are stored at $4 \pm 2^{\circ}\text{C}$ or $-15 \pm 5^{\circ}\text{C}$.

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Acetonitrile Extraction of Burn Pan Residue Samples and Handling of Extracts for SFC/MS and SFC/TEA

A. Extraction of Burn Pan Residue Samples

1. Following methylene chloride (MeCl_2) extraction (LOI-008), each residue sample, including matrix spikes and duplicates, is spiked with 2 μg of 9-phenyl anthracene (internal standard) and extracted with 700 mL of HPLC grade (or better) acetonitrile (ACN) in a 500 mL Soxhlet extractor as per LOI-009 for 6 h at a solvent temperature of 80 to 81°C.
2. For every extraction event, a method blank consisting of 3 clean filters is extracted.

B. Handling of Extracts

1. One third of the (MeCl_2) extract (LOI-008) is concentrated to approximately 1 mL and the ACN extract is concentrated to approximately 1 to 2 mL volume using a rotary evaporator (<28°C) as per LOI-010.
2. The concentrated (MeCl_2) aliquot is eluted with MeCl_2 through solid phase extraction tubes (Alltech Sample Preparation Silica, 30 to 70 μm , 60Å) until a total volume of 4 mL is collected in a vial and concentrated to 0.2 mL under N_2 purge.
3. One third of the ACN extract is then eluted through the solid phase extraction tube with ACN until a total volume of 4 mL is collected and concentrated to 0.2 mL under N_2 purge.
4. The concentrated MeCl_2 and ACN extract aliquots are combined in an amber vial and spiked with 1 μg of 1-Nitronaphthalene- d_7 (internal standard) for SFC/MS and SFC/TEA analyses.
5. The remaining two thirds of extract are archived in an amber vial with a Teflon lined cap.

C. Storage of Extracts

Vials containing the residue extracts are stored at $4 \pm 2^{\circ}\text{C}$ or $-15 \pm 5^{\circ}\text{C}$.

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Methylene Chloride Extraction of Burn Pan and Detonation Residue Samples and Handling of Extracts for GC/MS, SFC/MS, and SFC/TEA Analyses

A. Extraction of Burn Pan and Detonation Residue Samples

1. A representative aliquant (up to 50g) of each residue sample is carefully wrapped in a clean filter to prevent particulate loss, spiked with 8 μ g of base and neutral surrogates (nitrobenzene-d₅, 2-fluorobiphenyl, and terphenyl-d₁₄) and 16 μ g of acid surrogates (2-fluorophenol, phenol-d₆, and 2,4,6-tribromophenol), and extracted with 700 mL of HPLC grade (or better) methylene chloride (MeCl₂) in a 500 mL Soxhlet extractor as per LOI-009 for 6 h at a solvent temperature of 39 to 40°C.
2. For every extraction event, a method blank consisting of 3 clean filters is extracted.
3. For every 20 samples extracted, a matrix spike (MS) is prepared and extracted. A MS for GC/MS consists of a second aliquant of one residue sample spiked with 20 μ g each of target analytes listed in U.S. EPA SW-846 Method 8270A dissolved in an appropriate solvent. A MS for SFC/MS & SFC/TEA consists of a second aliquant of one residue sample spiked with 4 μ g each of target analytes in table 5.1 (LOI-005).
4. For every 20 samples extracted, a duplicate is prepared and extracted. A sample duplicate consists of a separate aliquant of one residue sample prepared and extracted in the same way as the sample aliquant.

B. Handling of Extracts

1. One third of the extract is transferred to a certified clean amber jar with a Teflon lined cap for preparation (LOI-007) prior to SFC/MS and SFC/TEA analysis.
2. The remaining two thirds of the extract are filtered through approximately 7 g of anhydrous sodium sulfate.

3. The filtered extract is concentrated in a Kuderna-Danish apparatus to approximately 1 to 2 mL volume (70 to 80°C) as per U.S. EPA SW-846 Method 3500.
4. The concentrated extract is split into 2 aliquots.
5. One aliquot is archived in an amber vial with a Teflon lined cap.
6. The other aliquot is concentrated to 0.2 mL under N₂ purge (21 to 24°C).

C. Storage of Extracts

Vials and bottles containing the residue extracts are stored at or below $4 \pm 2^{\circ}\text{C}$ or $-15 \pm 5^{\circ}\text{C}$.

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Soxhlet Extractor Procedure

A 500 mL Soxhlet extractor is used to extract samples. A 1700 mL Soxhlet extractor is used to extract all filters prior to sampling.

1. 700-800 mL of solvent are placed in a 1000 mL extraction flask with one boiling chip or 2300 mL in a 3000 mL extraction flask with several boiling chips.
2. The extraction flask is placed in the heating mantle.
3. The Soxhlet extractor is assembled with the filter sample or new filters in place.
4. Cold water to the condenser is turned on.
5. A variable auto transformer is connected to the heating mantle to control the temperature.
6. The heating mantle is heated to a temperature at which the solvent boils rapidly and smoothly and the Soxhlet turn-over time is approximately 10-15 min.
7. After extracting for the required time, the power is switched off, and the extractor is allowed to cool.
8. Any residual solvent in the Soxhlet tube is added to the solvent in the extraction flask.
9. When extracting a sample, the contents of the extraction flask are saved for concentration and subsequent analysis.
10. After extraction, all parts of the apparatus that come into contact with the extract are cleaned.

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Concentration by Rotary Evaporation

The rotary evaporator is used to minimize volatile analyte losses while concentrating large volumes of sample extract solution.

1. The condenser cooling water is turned on.
2. The vacuum stopcock is turned so that no vacuum is created (aligned with the vacuum release orifice).
3. The round bottom evaporating flask containing the sample extract is connected to the exposed end of the vapor duct.
4. The evaporating flask is lowered so that it is partially submerged in the water bath, which is maintained at or below 28°C.
5. The rotation speed of the drive unit is adjusted to approximately 80 rpm and the sample extract is put under vacuum by rotating the vacuum stopcock one quarter turn. Care should be taken that no bumping or foaming occurs.
6. The liquid volume is reduced by evaporation to 1 to 2 mL, the vacuum is released, and the drive unit is stopped. The evaporating flask is removed. The receiving flask is emptied in the appropriate waste container.
7. The concentrated sample extract is transferred to a glass vial with a Teflon lined cap and the round bottom flask is rinsed with approximately 0.5 mL of solvent two times transferring each rinse to the vial.
8. The vapor duct is cleaned before concentrating the first extract and in between different kinds of extracts. The following procedure is followed:
 - a. With the vacuum off, the vapor duct is rotated at a slow speed.

- b. A 100 mL beaker of appropriate solvent is held under the rotating vapor duct. All of the ground glass joint is wetted by the solvent.
- c. The beaker of solvent is lowered until part of the opening of the vapor duct is above the surface of the solvent.
- d. The vacuum stopcock is closed.
- e. The beaker is raised and lowered gradually to allow the duct to suck up enough solvent to coat the end of the tube.
- f. The vacuum stopcock valve is opened partially; the rotating vapor duct is submerged in the beaker of solvent and the stopcock valve is adjusted until the vapor duct fills with solvent (but does not overflow). The solvent is allowed to remain in the rotating tube for at least 5 s. The stopcock is closed and some of the solvent is allowed to overflow into the rotary evaporator solvent receptacle.
- g. The receiving flask is emptied.
- h. After every twenty samples, vapor duct washings are collected and analyzed for target analytes.

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Supercritical Fluid Chromatography/Mass Spectrometry (SFC/MS)

This LOI describes the SFC/MS instrumentation and methodology for sample extract analyses. Extract preparation is described in LOIs-005 and 007. Two complementary SFC/MS analyses are performed on each extract. Negative chemical ionization (NCI) is used to analyze nitroaromatics, nitramines, nitrate esters, and most nitroso compounds. Positive chemical ionization (PCI) is used for polynuclear aromatic hydrocarbons (PAHs), their nitrogen and oxygen heterocyclic counterparts, amines, nitroso amines, and other compounds that form positive ions. Target analytes are analyzed by selected ion monitoring (SIM).

A. Analysis by NCI/SIM/MS

1. The instrument used for this method is a modified Lee Scientific Model 600/supercritical fluid chromatograph Finnegan-MAT Incos 50 mass spectrometer. Inserted between the mass analyzer and the turbo molecular pump is an Alpine West Laboratories (AWL) liquid nitrogen cooled cryogenic pump. The glass barrel in which the mass analyzer resides is wrapped with heat tape and insulation and heated to $\sim 235^{\circ}\text{C}$. The chromatograph is connected to the mass spectrometer by an AWL direct probe interface. The interface is heated to 160°C except the probe tip, which is heated to 300°C . An AWL solid phase injector is used for sample introduction into the super fluid chromatograph.
2. The instrument is mass calibrated and tuned, as necessary, using perfluorotributylamine (FC-43). Mass calibration is performed using software and recommended procedures provided by the manufacturer.
3. Chromatographic separation takes place inside the chromatograph. A $0.1\text{ }\mu\text{m}$ film of 25% cyanobiphenylpolysiloxane stationary phase chemically bonded in a $0.025\text{-mm i.d.} \times 5\text{ m}$ fused silica capillary column is used for separation. A restrictor, housed in the interface, delivers the SFC eluent to the spectrometer. The initial fluid density and temperature are sample dependent. Density and temperature programming are used for analysis. Conditions (SFC and MS) are identical for standard and sample analyses.

4. Data acquisition is accomplished by the mass spectrometer and consists of repetitive scanning of preselected m/e ratios that are characteristic of the target analytes. Cycle time is approximately 1 s. Data acquisition begins with and continues throughout the chromatographic separation process.
5. A standard solution (calibration standard) of target analytes is analyzed at the beginning of every day and after every 10 samples.
6. Analytical blanks (calibration blanks) are analyzed with the samples.
7. A response curve is generated by analysis of the standard solutions at 0.1, 0.5, 1.0, 5.0, 7.0, and 10 ng μL^{-1} of each compound (LOI-014, table 14.1). The internal standard is present in each standard at 15 ng μL^{-1} . The response curve consists of the response of a particular analyte relative to that of the internal standard, plotted as a function of analyte concentration. The purpose of the response curve is to provide quality assurance of response linearity.
8. Response factors are established for each analyte (I) from analyses of a standard solution. The response factor (RF_i) is defined as follows:

$$\text{RF}_i = \frac{(A_i)(C_{\text{IS}})}{(A_{\text{IS}})(C_i)} \quad (1)$$

where A_i and A_{IS} are the selected ion chromatographic peak areas of the analyte and internal standard, respectively. C_i and C_{IS} are the concentrations of the analyte and internal standard, respectively.

9. Samples are analyzed using the same instrumental conditions as the standard. Identification of target analytes is by retention time and response at the specific masses monitored for each analyte. The concentration of each analyte (C_i) as mass per unit volume of extract (eg. ng/mL) is defined as:

$$C_i = \frac{(A_i)(C_{\text{IS}})}{(A_{\text{IS}})(\text{RF}_i)} \quad (2)$$

where the variables are functionally equivalent to those in Section A.8 but whose values are obtained from the analytical data of the sample. Thus, sample target analyte concentrations are calculated from relative response factors of sample and standard solutions.

B. Analysis by PCI/SIM/MS

1. The instrument used for this method is a Lee Scientific Model 600/supercritical fluid chromatograph Finnegan-MAT Incos 50 mass spectrometer.
2. The instrument is mass calibrated, tuned, and operated as in Sections A.2 to A.9 except that the MS is tuned and operated in the positive chemical ionization mode.

C. Analysis by NCI/FS/MS

1. The instrument used for this method is a Lee Scientific Model 600/supercritical fluid chromatograph Finnegan-MAT Incos 50 mass spectrometer.
2. The instrument is mass calibrated, tuned, and operated as in Sections A.2 and A.3.
3. Data acquisition consists of repetitive scanning from m/e 100 to m/e 500 with a cycle time of approximately 1 s. Data acquisition begins with and continues throughout the chromatographic separation process.
4. The remainder of the analytical procedure is equivalent to that described in Sections A.5 to A.9.

D. Analysis by PCI/FS/MS

1. The instrument used for this method is a Lee Scientific Model 600/supercritical fluid chromatograph Finnegan-MAT Incos 50 mass spectrometer.

2. The instrument is mass calibrated, tuned, and operated as in Sections C.2 to C.4, except that tuning is conducted in the positive chemical ionization mode.

E. Reporting and Archiving of Data

1. The results from each SFC/MS analysis consist of the following:
 - (a) A reconstructed (total ion) chromatogram.
 - (b) A mass chromatogram containing the quantitation area for each analyte and internal standard.
2. For each sample, a summary table is provided containing all target analytes found with final concentrations expressed as ng/sample.
3. Raw SFC/MS data are archived in disk image format on data cartridge tapes (DC 300 XL/P) at 10,000 ft pi.

F. Determination of Calibration Curves and Instrument Detection Limits for SFC/MS

1. A stock solution of the semivolatile organic target analytes in acetonitrile at a concentration of $100 \text{ ng } \mu\text{L}^{-1}$ is prepared.
2. Logarithmic (1 to 10, 1 to 100, etc.) dilutions of the stock solution are analyzed until observed analyte peak areas in the reconstructed total-ion chromatogram are less than three times that of the background signal. The INCOS 50 data system reports the peak areas in relative ion counts (RIC).
3. The analyte concentration at which the RIC is three times that of the background corresponds to the instrumental detection limit.
4. A calibration curve consisting of a graph of concentration versus observed peak area is constructed for each analyte. This serves as a standard curve and is sometimes used to determine concentrations in unknown samples. This is accomplished by matching the observed signal to the corresponding concentration for that analyte. Where possible all

quantitations are determined from response factors and comparisons to the internal standards.

5. All of the compounds on the semivolatile organic target analyte list are tested in a like manner.

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Supercritical Fluid Chromatography/ Chemiluminescence Detection (SFC/TEA)

This LOI describes the SFC/TEA instrumentation and methodology for analysis of sample extract. Extract preparation is described in LOIs-005 and 007. The TEA detector is a sensitive nitrosyl-group specific detector. Therefore, it is very useful in the analysis of nitroso and nitro compounds and nitrate esters. Table I is a list of some compounds detected by the detector. At 800°C it detects virtually all of these compounds. At 400°C, nitroso compounds, nitramines, and nitrate esters are detected, but nitro aromatics are not. Untargeted analytes detected by SFC/TEA can be analyzed by SFC/FS/MS (LOI-011) for identification purposes.

A. Method of Analysis

1. The instrument used for this method is a Lee Scientific Model 600 supercritical fluid chromatograph/thermodynamics TEA Model 543 analyzer. The interface is a tube heated to 350°C. An AWL solid phase injector is used for sample introduction.
2. Chromatographic separation takes place inside the chromatograph. A 0.1 μm film of 25% cyanobiphenylpolysiloxane stationary phase chemically bonded in a 0.050 mm i.d. x 6 m fused silica column is used for separation. A frit restrictor delivers the SFC eluent to the detector. The initial fluid density and temperature are sample dependent. Density and temperature programming are used for analysis. Conditions (SFC and TEA) are identical for standard and sample analyses.
3. Data acquisition is accomplished by a Spectra-Physics Model SP 4270 integrator, which records the detector output signal together with peak retention times and areas.
4. A standard solution of target analytes (calibration standard) is analyzed at the beginning of each day and after every 10 samples.
5. Analytical blanks (calibration blanks) are analyzed with the samples.
6. A response curve is generated by analysis of the standards at 0.5, 1.0, 5.0, 7.0, and 10 $\text{ng } \mu\text{L}^{-1}$ of each compound (LOI-005, table 5.1). The internal

standard is present in each standard at $5 \text{ ng } \mu\text{L}^{-1}$. The response curve consists of the response of a particular analyte relative to that of the internal standard, plotted as a function of analyte concentration. The purpose of the response curve is to provide quality assurance of response linearity.

7. Response factors are established for each analyte (I) from analyses of a standard solution. The response factor (RF_i) is defined as follows:

$$\text{RF}_i = \frac{(A_i)(C_{\text{IS}})}{(A_{\text{IS}})(C_i)} \quad (1)$$

where A_i and A_{IS} are the chromatographic peak areas of the analyte and internal standard, respectively. C_i and C_{IS} are the concentrations of the analyte and internal standard, respectively.

8. Samples are analyzed using the same instrumental conditions as the standard. Identification of target analytes is by retention time. The concentration of each analyte (C_i) as mass per unit volume of extract (eg. ng mL^{-1}) is defined as:

$$C_i = \frac{(A_i)(C_{\text{IS}})}{(A_{\text{IS}})(\text{RF}_i)} \quad (2)$$

where the variables are functionally equivalent to those in Section A.7 but whose values are obtained from the analytical data of the sample. Thus, sample target analyte concentrations are calculated from relative response factors of sample and standard solutions.

B. Reporting of Data

1. In contrast to the mass spectrometer, the chemiluminescence detector is not capable of resolving overlapping peaks and quantitation is dependent upon chromatographic resolution (the separation of peaks in the chromatogram). The results from each SFC/TEA analysis consist of the following:

- (a) A chromatogram.
 - (b) A table of peak retention times, areas, and relative peak areas.
2. For each sample, a summary table is provided containing all target analytes found with final concentrations expressed as ng/sample.

C. Determination of Calibration Curves and Instrument Detection Limits for SFC/MS

- 1. A stock solution of the semivolatile organic target analytes in acetonitrile at a concentration of $100 \text{ ng } \mu\text{L}^{-1}$ is prepared.
- 2. Logarithmic (1 to 10, 1 to 100, etc.) dilutions of the stock solution are analyzed until the observed detector signal for each analyte is less than three times the background signal.
- 3. The analyte concentration at which the signal is three times that of the background corresponds to the instrumental detection limit.
- 4. A calibration curve consisting of a graph of concentration versus observed peak area is constructed for each analyte. This serves as a standard curve and is sometimes used to determine concentrations in unknown samples. This is accomplished by matching the observed signal to the corresponding concentration for that analyte. Where possible, all quantitations are determined from response factors and comparisons to the internal standards.
- 5. All of the compounds on the semivolatile organic target analyte list are tested in like manner.

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Table 12-1. Some compounds that can be detected by the TEA.

N-Nitrosodiethanolamine	
N-Nitrosodiethylamine	2-Nitronaphthalene
N-Nitrosodimethylamine	1-Nitropyrene
N-Nitrosodiphenylamine	1,3,5-Trinitrobenzene
N-Nitrosodipropylamine	2,4,6-Trinitrotoluene
N-Nitroso-N-ethylaniline	Other nitroaromatics
N-Nitrosomorpholine	N-Methyl-N-2,4,6-tetranitroaniline
N-Nitrosornicotine	2-Nitrodiphenylamine
N-Nitrosopiperidine	Other nitroanilines
N-Nitrosopyrrolidine	Hexahydro-1,3,5-trinitro-1,3,5-triazine
Nitrosoalkanes	Octahydro-1,3,5,7-tetranitro-1,3,5,7-
Nitrosoaromatics	tetrazocine
Nitroalkanes	Other nitramines
2,4-Dinitrotoluene	Nitrite esters
2,6-Dinitrotoluene	Ethylene glycol dinitrate
1-Nitronaphthalene	Isosorbitol dinitrate
	Nitroglycerin
	Pentaerythritol tetranitrate

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Gas Chromatography/Mass Spectrometry (GC/MS)

This LOI describes the GC/MS instrumentation and methodology for analysis of sample extracts. Extract preparation is described in LOIs-006 and 008. The GC/MS analysis is performed according to USEPA SW-846 Method 8270. Full scan electron ionization (EI/MS) is used to detect and quantitate the target analytes. A library search using the NBS library of compounds is conducted to identify untargeted analytes that may be of interest.

A. Analysis by EI/MS

1. The instrument used for this method is a Hewlett Packard 5890/5970 gas chromatograph/mass spectrometer operated in a conventional GC/MS mode.
2. The instrument is mass calibrated and tuned every 12 hour shift using decafluorotriphenylphosphine (DFTPP). Mass calibration is performed using software and recommended procedures provided by the manufacturer.
3. Chromatographic separation involves an HP 5890 gas chromatograph. A fused silica capillary transfer line delivers the eluent from the GC column to the mass spectrometer. One microliter of sample is injected into the heated (270°C) split less injection port. A Restek XTI-5 (30 m, 1 μ m film, 0.25 mm i.d.) fused silica capillary column is used for separation. Temperature programming (50°C hold 4 min. then 10°C/min. to 315°C) is used for analysis, and GC/MS conditions are identical for standard and sample analyses.
4. Data acquisition consists of repetitive scanning from mass 35 to 500 with a cycle time of approximately 1 s. Data acquisition continues throughout the chromatographic separation process.
5. A standard containing the target analytes of interest for a specific test is analyzed every 12 hour shift in which samples are analyzed.

6. A method blank is extracted and analyzed with every batch of 20 or fewer samples.
7. From the analyses of standard target analyte solutions (5, 25, 40, 60, and 80 ng μL^{-1}) containing internal standards (per SW-846 Method 8270), response factors are measured at the various concentrations. The response factor (RF_i) is defined as follows:

$$\text{RF}_i = \frac{(A_i)(C_{\text{IS}})}{(A_{\text{IS}})(C_i)} \quad (1)$$

where A_i and A_{IS} are the chromatographic peak areas of the analyte and internal standard, respectively. C_i and C_{IS} are the concentrations of the analyte and internal standard, respectively. An average response factor (RF_{AVE}) is determined for each analyte relative to the response of its internal standard. The linearity of the RF_{AVE} of each target analyte over the range of standard concentrations is determined by the percent relative standard deviation (%RSD) of the response factors for each analyte.

8. Samples are analyzed using the same instrumental conditions as the standard. Identification of target analytes is by retention time and mass spectra. The concentration of each analyte (C_i) as mass per unit volume of extract (eg. ng/mL) is defined as:

$$C_i = \frac{(A_i)(C_{\text{IS}})}{(A_{\text{IS}})(\text{RF}_{\text{AVE}})} \quad (2)$$

where the variables are functionally equivalent to those in Section A.7 but whose values are obtained from the analytical data of the sample. Thus, sample target analyte concentrations are calculated from relative response factors of sample and standard solutions.

9. Mass spectral interpretation of all target analytes and up to 25 nontarget analytes determined above the method detection limit is performed.

B. Data Reporting and Archiving

1. The results from each EI/MS analysis consist of the following:
 - (a) A total ion chromatogram annotated to indicate the peaks identified.
 - (b) A background corrected mass spectrum for each identified compound.
 - (c) A list of identified compounds with retention times and extract concentrations.
 - (d) A library search summary package with spectral information, approximate concentrations and tentative identification of non-target compound peaks (up to 25 total).
2. For each sample, an Analytical Report is provided containing all target analytes found with final concentrations expressed as ng/sample.
3. Raw GC/MS data are archived in exact binary images on 16 rack CS80 cartridge tapes or 9 track reel to reel tapes.

C. Determination of Calibration Curves and Instrument Detection Limits for GC/MS

1. Standard solutions are prepared according to Method 8270. Internal standards and surrogate compounds are spiked into all samples as per the method.
2. Five concentration levels (5, 25, 40, 60, 80 ng μL^{-1}) of calibration standard solutions were used to calibrate the instrument. A continuing calibration check is analyzed daily and all criteria checked prior to analyzing samples. The average response factor (RF_{AVE}) for each compound is used for quantitation.

3. The retention times and calibration are updated with the calibration check standard.
4. Method detection limits (MDL) are established experimentally by the method of 40 CFR 136, Appendix B, in accordance with Method 8270.
5. Analytes are quantitated as low as the MDLs.
6. All of the compounds on the semivolatile organic target analyte list are tested in a like manner.

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Semivolatile Organic Target Analyte Selection

A semivolatile organic target analyte list will be determined for each munition or propellant tested. Each list will contain the following:

1. The target analyte list for TNT (Table 1).
2. The explosive or propellant compound tested.
3. Nitroso and nitro compounds and nitrate esters identified by SFC/TEA and SFC/FS/MS.
4. Compounds identified by GC/EI/MS.
5. Surrogate compounds selected from "Literature Survey of Pyrolysis, Detonation, and Combustion of Explosives and Propellants" by W. H. Lee (Alpine West Laboratories, 25 Feb 1992).
6. Surrogate compounds projected as possible combustion products and residues, based on organic chemistry of explosives, propellants, and related substances.

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Table 14.1. Target Analyte List for TNT

-
- | | |
|-----|------------------------|
| 1. | 2,6-Dinitrotoluene |
| 2. | 2,4-Dinitrotoluene |
| 3. | 2-Nitronaphthalene |
| 4. | 2,4,6-Trinitrotoluene |
| 5. | 2-Nitrodiphenylamine |
| 6. | 1,3,5-Trinitrobenzene |
| 7. | 1-Nitropyrene |
| 8. | Naphthalene |
| 9. | Phenol |
| 10. | Dibenzofuran |
| 11. | N-Nitrosodiphenylamine |
| 12. | Diphenylamine |
| 13. | Pyrene |
| 14. | Benz[a]anthracene |
| 15. | Benzo[a]pyrene |
-

Sampling and Determination of Particulate Matter as PM₁₀

This LOI Describes the equipment and methodology for the sampling and determination of particulate matter as PM₁₀.

A. Sample Collection by the PM₁₀ system

1. A General Metal Works Model GUV-15H PM10 Size Selective High Volume Sampler is used to collect samples. The system consists of a size selective inlet mounted on a basic high volume air sampler. The system, also, includes a volumetric flow controller, a digital timer, and a flow recorder.
2. The system automatically discards particles larger than 10 μm and collects particles 10 μm and smaller, while maintaining a constant air sampling flow of 40 cfm.

B. Determination of Weight of PM₁₀ Particle Matter

1. The filters are prepared and handled according to LOI-002.
2. The filters are weighed prior to and following sampling as described in LOI-002.
3. The weight of collected PM₁₀ particulate matter is calculated by subtracting the pre-sampling filter weight from the post sampling filter weight.

C. Reporting of Data

A summary table is provided listing the total weight of PM₁₀ particulate matter collected in each sample.

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Digestion of Quartz Fiber Filter Samples for ICP

A. Digestion of Quartz Fiber Filter Samples

1. Samples are digested according to U.S. EPA SW-846 Method 3050A. Four circular sections of filter are placed into a 250 mL beaker. Five mL of 1:1 HNO_3 are added and the slurry is mixed and covered with a watch glass. The sample is heated to 95°C for 10 minutes (without boiling) and allowed to cool. Two and one half mL of concentrated HNO_3 are added, the watch glass is replaced, and the slurry is refluxed until the volume is reduced to 3 mL.
2. After cooling, 1 mL of deionized water and 1.5 mL of 30% H_2O_2 are added. The sample is heated cautiously until effervescence subsides. No more than a total of 5 mL of 30% H_2O_2 is used.
3. Five mL of conc HCL and 5 mL deionized water are added and the sample is heated for an additional 15 minutes. It then is allowed to cool.
4. The solution is filtered through a Whatman No. 41 filter paper into a 100 mL volumetric flask. The beaker, residue, and filter medium are washed thoroughly with deionized water. The volume is adjusted to 50 mL and the solution is transferred to a 125 mL container.
5. For every 20 samples digested, a laboratory control sample (LCS), a reagent blank, a duplicate sample, and a matrix spike are digested.
6. The LCS consists of 0.5 g of a standard soil sample.
7. The matrix spike consists of 4 filter sections spiked with the target analytes.

B. Handling of Digestates

The digestate is analyzed by ICP as per LOI-018.

C. Storage of Digestates

The capped digestate containers are stored at room temperature. Digestates can be stored up to 6 months from the time of sample collection.

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Digestion of Quartz Fiber Filter Samples for CVAA

A. Digestion of Quartz Fiber Filter Samples

1. Two circular sections of filter are placed into a 250 mL beaker to which 5 mL of H_2SO_4 and 2.5 mL of HNO_3 are added. The contents of the beaker are mixed and heated for two minutes in a water bath at 95°C . The sample is removed from the heat and allowed to cool.
2. Fifty mL of deionized water and 15 mL of 5% KMnO_4 solution are added and the slurry is mixed. Additional portions of 5% KMnO_4 solution are added, if necessary, until a purple color persists for at least 15 minutes. Eight mL of 5% $\text{K}_2\text{S}_2\text{O}_8$ solution are added and the slurry is mixed.
3. The sample is heated for 30 minutes in a water bath at 95°C and cooled.
4. Six mL of sodium chloride/hydroxylamine hydrochloride solution are added 6 mL increments until KMnO_4 is completely reduced. The volume is adjusted to 100 mL with deionized water, and mixed.
5. For every 20 samples digested, a laboratory control sample (LCS), a reagent blank, a duplicate sample, and a matrix spike sample are digested.
6. The LCS consists of 0.2 g of a standard soil sample.
7. The matrix spike consists of 2 filter sections spiked with the target analyte.

B. Handling of Digestates

1. The digestate is analyzed by the cold vapor technique as per LOI-019.
2. The digestate must be analyzed within 24 h.

C. Storage of Digestates

The capped digestate containers are stored at room temperature.

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Inductively Coupled Plasma Optical Emission Spectrometry

This LOI describes the ICAP™ instrumentation and methodology for sample digestate analyses. Filter preparation is described in LOI-016. The analysis is performed according to USEPA SW-846 Method 6010A. Prepared samples are nebulized and the resulting aerosol is introduced into the plasma. Spectral emission is induced by the interaction between the energetic argon atoms and the elements in the samples. The characteristic emissions are then measured and compared to the responses of known concentrations. After compensation for background and spectral interferences, the concentration of the sample can then be calculated.

A. Analysis by ICP

1. The instrument used for this method is a Thermo Jarrell Ash ICAP™ 61E.
2. The instrument is calibrated according to the manufacturer's recommendations with two standard solutions (a high concentration calibration standard and a calibration blank).
3. Standard solutions of target analytes are analyzed with the sample to verify instrument calibration.
4. Reagent blanks are analyzed before analyzing any samples and after every 10 samples.
5. Analyses are performed in the linear range of the response curve.

B. Reporting and Archiving of Data

1. The ICP provides a real-time instrument printout for each analyte.
2. A summary of all target analyte concentrations is available for each analytical run.
3. Raw ICAP data are archived to disk.

Mountain States Analytical, Inc.

LOI-018, Rev. 1

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Cold Vapor Atomic Absorption Spectrometry

This LOI describes the CVAA instrumentation and methodology for sample digestate analyses. Filter preparation is described in LOI-017. The reaction for the mercury analysis is a simple reduction reaction. The mercury (Hg^{+2}) is reduced with stannous (Sn^{+2}) chloride to liberate mercury metal and Sn^{+4} . The sample is made acidic with hydrochloric acid to maintain the reducing environment of the reaction. Inert gas sweeps the volatile mercury into the absorption cell in the optical path of the atomic absorption spectrophotometer.

A. Analysis by CVAA

1. The instrument used for this method is a Varian SpectrAA-20 Atomic Absorption Spectrometer with a VGA-76 Vapor Generation Accessory.
2. The instrument is calibrated according to the manufacturers recommendations, using 5 concentration levels of mercury standard solutions.
3. Standard solutions of target analytes are analyzed before analyzing samples and after every 10 samples to verify instrument calibration.
4. Reagent blanks are analyzed before analyzing samples and after every 10 samples.
5. Analyses are performed within the range of the calibration curve.

B. Reporting and Archiving of Data

The instrument provides a real-time printout.

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Quality Control Plan: Procedures for Accuracy, Precision, and Completeness

A. Accuracy, Precision, and Completeness

1. Accuracy is measured by the agreement of the result of an analyzed standard with the known value. It is usually reported in terms of percent recovery (%R).

$$\%R = \frac{\text{result}}{\text{true value}} * 100\%$$

2. Precision is measured by agreement of replicate measurements of a standard or sample. Duplicate precision is reported in terms of relative percent difference (RPD), and precision of multiple replicates is reported in terms of percent relative standard deviation (%RSD).

$$RPD = \frac{X_1 - X_2}{\left(\frac{X_1 + X_2}{2} \right)} * 100 \qquad \%RSD = \frac{SD_{n-1}}{\text{mean}} * 100\%$$

3. Completeness is measured as percent of samples analyzed versus the number of samples collected. Completeness is reduced by loss of a sample, invalidation of a result, and failure to analyze a sample.

B. Sample Storage

1. All samples are stored at $4 \pm 2^\circ\text{C}$ or $-15 \pm 5^\circ\text{C}$.
2. The temperatures of the refrigerators and freezers used for sample storage are monitored and recorded daily.
3. Any temperature excursion is corrected promptly or samples are transferred to a properly functioning refrigerator or freezer.

C. Weighing

1. All balances are calibrated yearly by a service technician using NIST traceable standard weights.
2. The analytical balances (Mettler Toledo AB104 and Fisher AX200H) are accurate to ± 0.0002 g. The top loading balance (Fisher XL-300) is accurate to ± 0.002 .
3. Balances are tested for accuracy using standard weights at frequent intervals (LOI-002).

C. Extraction

1. One filter blank is extracted for every ten to twenty filter samples.
2. Efficiency (accuracy) of extraction is measured by re-extraction of one or more samples and by recoveries of surrogate standards added to samples before extraction.

D. SFC/MS and SFC/TEA

1. Instrument calibrations are evaluated for accuracy by analyzing a calibration standard at the beginning of every analysis run and after every 10 samples. The acceptance criterion for calibration response factor percent difference is $< 50\%$.
2. Standard target solutions are run with samples and standard recoveries are evaluated against acceptance criteria of 50% to 150%.
3. Filter blank extracts are analyzed at a frequency of one in ten to one in twenty samples.
4. A random number of extracts in every set is analyzed in duplicate to evaluate measurement precision. The RPD must be less than 50.

E. GC/MS

1. Instrument calibrations are evaluated for accuracy by analyzing a calibration standard at the beginning of every analysis run and after every 10 samples. The acceptance criterion for calibration response factor percent difference is <30%.
2. Analyte standard solutions are run with samples and standard recoveries are evaluated against acceptance criteria specific to each analyte as given in USEPA SW-846 Method 8270.
3. Filter blank extracts are analyzed at the frequency of no greater than one in twenty samples. Target analytes must be less than the quantitation limits.
4. A random number of extracts in every set is analyzed in duplicate to evaluate measurement precision. The RPD must be less than analyte-specific limits given in USEPA SW-846 Method 8270.

F. Metals (ICP and CVAA)

1. Instrument calibrations are evaluated for accuracy by analyzing a calibration standard at the beginning of every analysis run and after every 10 samples. The acceptance criteria for standard recoveries are 90% to 110%.
2. Analyte standard solutions are run with samples and standard recoveries are evaluated against acceptance criteria of 80% to 120%.
3. Method blanks are analyzed at the frequency of one per set of 20 (or fewer) samples. Target analytes must be less than the quantitation limits.
4. Matrix spikes are analyzed at a frequency of one per set of 20 (or fewer) samples. All analytes must be recovered within 80% to 120%.
5. A random number of extracts in every set is analyzed in duplicate to evaluate measurement precision. The RPD must be less than 20.

G. Ion Chromatograph

1. Instrument calibrations are evaluated for accuracy by analyzing a calibration standard at the beginning of every analysis run and after every 10 samples. The acceptance criteria for standard recoveries are 90% to 110%.
2. Analyte standard solutions are run with samples and standard recoveries are evaluated against acceptance criteria of 80% to 110%.
3. Filter blank extracts are analyzed at the frequency of one in ten to one in twenty samples. Target analytes must be less than the quantitation limits.
4. A random number of extracts in every set is analyzed in duplicate to evaluate measurement precision. The RPD must be less than 20.

H. GC/ECD

1. Instrument calibrations are evaluated for accuracy by analyzing a calibration standard at the beginning of every analysis run and after every 10 samples. The acceptance criterion for calibration response factor percent difference is <15%.
2. Filter blank extracts are analyzed at the frequency of no greater than one in twenty samples.
3. A random number of extracts in every set is analyzed in duplicate to evaluate measurement precision. The RPD must be less than 30.

I. Sample Handling

1. Completeness of sample analyses is ensured by laboratory practices that account for every sample and analysis.
 - a. A chain of custody procedure for tracking samples is in place.
 - b. Each sample is given an identification number.

- c. All samples are logged according to identification number, treatment, and location.
- d. A sample custody transfer form is used to transfer samples between MSAI and other facilities.

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Preparation of BangBox® Fabric Swatch Samples

When munitions containing chlorine are burned or detonated in the BangBox®, significant quantities of HCl and/or chlorinated organic compounds may adsorb to the BangBox® wall.

Swatches of BangBox® fabric are hung in the BangBox® during tests of specific materials to determine losses of HCl and chlorinated organics to the BangBox® wall.

A. Swatch Construction

1. Clean fabric and swatches are handled only with clean cotton arm long gloves or clean forceps.
2. Rectangular pieces are cut from BangBox® fabric, 2.5 by 5 in. pieces from old fabric and 3 by 6 in pieces from new fabric.
3. Fabric pieces are washed in deionized water for 2 h. The container holding the fabric pieces is shaken at least 8 times and the water is changed at least 4 times during the 2 h. period.
4. Each fabric piece is supported separately on edge in a forced air oven and dried at 40°C for a minimum of 4 h.
5. Fabric pieces are sewn together in pairs, old to old and new to new. Pairs are sewn together such that the surfaces characteristic of the outside BangBox® well are flat against each other and only the surfaces characteristic of the inside BangBox® wall are exposed.

B. Swatch Preparation for Sampling

1. Clean swatches are handled only with clean cotton or nylon gloves or clean forceps.
2. Swatches are cleaned and dried by the procedure outline in Section A.3 and A.4.

3. Clean swatches are stored at ambient temperature in clean capped bottles.
4. Only swatches that have been extracted in water (LOI-028) are cleaned and used again. Swatches extracted in organic solvent (LOI-029) are not used again for sample collection.

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Extraction of BangBox® Fabric (Swatch) Samples for Anion Scan by Ion Chromatography and Handling of Extracts

A. Extraction of Swatch Samples

1. Each swatch sample is carefully placed into a 250 mL wide mouth glass jar (QC Clean).
2. To each jar containing a sample, 250 mL of deionized water are added. The jar is capped tightly and agitated every 10 to 15 minutes for 2 h or more.
3. The swatch is removed from the jar and the extract is submitted for analysis or stored.

B. Handling of Extracts

1. An aliquant of the extract is removed from the jar and placed in a micro disposable beaker.
2. The remaining sample is returned to cold storage.

C. Storage of Extracts

The extracts are stored at $4 \pm 2^{\circ}\text{C}$.

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Extraction of BangBox® Fabric (Swatch) Samples for Chlorinated Organics by GC/ECD and Handling of Extracts

A. Extraction of Swatch Samples

1. Each swatch sample is carefully placed into a 250 mL wide mouth glass jar (QC Clean).
2. To each jar containing a sample, 250 mL of hexane are added. The jar is capped tightly and agitated every 10 to 15 minutes for 2 h or more.

B. Handling of Extracts

1. A 10 mL aliquot of the extract is placed into a concentrator tube.
2. The extract is concentrated to a final volume of 1 mL.
3. The concentrated extract is placed into an autosampler vial for analysis by GC/ECD.

C. Storage of Extracts

Extracts are stored at $4^{\circ} \pm 2^{\circ}\text{C}$ or $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$.

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ION CHROMATOGRAPHY (IC)

This LOI describes the IC instrumentation and methodology for analysis of sample extract. Extract preparation is described in LOI-028. The IC uses ion exchange columns and a conductivity detector. Anions are separated based on their affinities for exchange sites of the resin. The anions (F^- , C^- , NO_3^- , NO_2^- , BR^- , SO_4^{2-} , & PO_4^{3-}) then elute at specific times and are detected by changes in conductivity.

A. Method of Analysis

1. The instrument used for this method is a Dionex DX-500 ion chromatograph/CD20 conductivity detector.
2. Separation takes place inside the column. The column (AS4A-SC) is composed of a 12 μm highly cross-linked polyethylvinylbenzene/divinylbenzene substrate agglomerated with anion exchange latex that has been completely aminated. The eluent used is 1.7 mM $NaHCO_3$ /1.8 mM Na_2CO_3 .
3. Data acquisition is accomplished by a 386 personal computer and stored on the hard drive and floppy disk.
4. A standard solution of target analytes is analyzed before any samples are analyzed and after every 10 samples.
5. Analytical blanks are analyzed before any samples and after every 10 samples.
6. Response curves are generated by analysis of standard solutions prepared at the concentrations listed in Table 30.1. The curves demonstrate linearity of instrument response to difference concentrations of the analytes in the standard solutions.
7. Samples are analyzed using the same instrumental conditions as the standards.

B. Reporting of Data

1. Results from each IC analysis consists of the following:
 - a. A chromatogram
 - b. A table of peak retention times, heights, and areas.
2. For each sample, a summary table is provided containing all target analytes found with concentrations expressed as mg/sample.

C. Determination of Calibration Curves

1. The response curves generated from the standard solutions listed in Table 30.1 are used as calibration curves.
2. The method detection limit (MDL) is determined by the method of 40 CFR 136, Appendix B.
3. Limit of Quantitation (LOQ) refers to a multiple (usually 5 to 10 times) of the MDL.

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**Table 30.1 Standard Solutions for Calibration
of the Ion Chromatograph**

Calibration Standard Analyte	<u>mgL⁻¹</u>	<u>mgL⁻¹</u>	<u>mgL⁻¹</u>	<u>mgL⁻¹</u>	<u>mgL⁻¹</u>	<u>mgL⁻¹</u>
F ⁻	0	0.1	0.4	1.0	2.0	4.0
Cl ⁻	0	0.5	1.0	2.0	4.0	8.0
Br ⁻	0	0.5	1.0	2.0	4.0	8.0
NO ₂ ⁻	0	0.08	0.61	1.22	2.44	3.05
NO ₃ ⁻	0	0.067	0.45	1.34	1.79	3.14
o-PO ₄ ⁻³	0	0.65	3.26	6.52	13.04	16.3
SO ₄ ⁻²	0	2.0	10.0	20.0	40.0	50.0

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Gas Chromatography/Electron Capture Detector (GC/ECD)

This LOI describes the GC/ECD instrumentation and methodology for analysis of sample extracts. Extract preparation is described in LOI-029. The GC/ECD analysis is performed according to USEPA SW-846 Method 8080. The electron capture detector is very sensitive to molecules with electronegative functional groups. (See Table 31.1.)

A. Method of Analysis by GC/ECD

1. The instrument used for this method is a Hewlett Packard 5890A GC/ECD.
2. Chromatographic separation involves an HP 5890 GC. A fused silica capillary transfer line delivers the eluent from the GC column to the detector. Two microliters of sample is injected into the heated (220 °C) split less injection port. The volatilized sample from the injection port is split evenly between two columns (a DB-608, fused silica capillary 30 m by 0.5 mm i.d. with 0.8 mm film thickness and a DB-1701 fused silica capillary 30 m by 0.5 mm i.d. with 1 mm film thickness) for chromatographic separation. Temperature programming is utilized: 80°C (4 min.) to 160°C (20° min.⁻¹) to 270°C (10° min.⁻¹). A separate detector is dedicated to each column. Conditions are identical for standard and sample analyses.
3. Data acquisition is accomplished by a personal computer with HP Enviroquant® software.
4. An instrument blank is analyzed approximately every 5 samples.
5. From the analyses of standard target analyte solutions (20, 100, and 500 µg L⁻¹) response factors are measured.
6. Samples are analyzed using the same instrumental conditions as the standard. Sample target analyte concentrations are calculated from relative response factors of sample and standard solutions.

B. Data Reporting and Archiving

1. The results from each GC/ECD analysis consist of the following:
 - (a) A chromatogram annotated to indicate the peaks identified.
 - (b) A background corrected quantitation worksheet for total identified chlorinated organics.
2. For each sample, an analytical report is provided containing target analyte found with final concentration expressed as $\mu\text{g}/\text{sample}$.
3. Raw GC/ECD data are archived.

C. Determination of Calibration Curves and Instrument Detection Limits for GC/ECD

1. The instrument is calibrated against a 3 point calibration curve using 20, 100, and 500 $\text{ng } \mu\text{L}^{-1}$ standard solutions. Calibration is tested every 20 samples with the mid-level standard solution (calibration check standard). The average response factor for each compound is used for quantitation.
2. The retention times and detector calibration are updated using the calibration check standard (if it passes all criteria).
3. Analytes are quantitated as low as the low calibration standard.
4. All of the samples are tested in a like manner.

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**Table 31.1 Electron Capture Detector Sensitivity to
Various Classes of Compounds**

<u>Compound Class</u>	<u>Relative Sensitivity</u>
Hydrocarbons	1
Ethers, esters	10
Aliphatic Alcohols, ketones, amines; mono-Cl, mono-F compounds	100
Mono-Br, di-Cl and di-F compounds	1000
Anhydrides and tri-Cl compounds	10^4
Mono-I, di-Br and nitro compounds	10^5
Di-I, tri-Br, poly-Cl and poly-F compounds	10^6

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THERMAL-OPTICAL ANALYSIS FOR ORGANIC/ ELEMENTAL CARBON AEROSOL

A. Analysis Description

The current instrument at Sunset Laboratory is capable of analyzing aerosol particles collected on quartz fiber filters for both organic carbon and elemental carbon. As with all current methods, it can only be referred to as a method-defined analysis. However, it was designed to specifically address some of the problems seen by other methods in assigning carbon to either the organic or elemental fraction. By careful control of operating conditions and constant monitoring of the optical absorbance of the sample during the analysis, this method is able to both prevent any undesired oxidation of original elemental carbon and make corrections for the inevitably-produced carbon char formed by the paralytic conversion of some organics into additional elemental carbon.

The instrument is similar to that described previously (An Automated Thermal-optical Method for the Analysis of Carbonaceous Aerosol: Richard L. Johnson, J. J. Shah, Robert A. Cary, and James J. Huntzicker, in *ACS Symposium Series, No. 167, Atmospheric Aerosol: Source/Air Quality Relationships*, Edward S. Macias and Philip K. Hopke, Eds. (1981). However there are certain modifications, one being that the optical darkness of the filter is monitored by transmitting the laser light through the sample filter rather than by observing the back-scattered light off the surface containing the sample deposit. Another modification is that different temperature steps are used in order to allow for the detection of inorganic carbonates which may be present. As with the previously described system, the final part of the analysis is the injection of a known amount of methane which serves as an internal quantifying standard as well as a quality assurance step.

A brief description of the procedure follows:

1. In a completely oxygen-free helium atmosphere, the sample is heated in four temperature steps to remove all the organic carbon present. The transition from the third temperature to the fourth temperature (from 500°C to 650°C) will quickly decompose inorganic carbonates.

producing a sharp, indicative peak. During this first phase, there are nearly always some organic compounds which are paralytically converted to elemental carbon, sometimes as high as 30%. This is continuously monitored by measuring the transmission of a He-Ne laser through the filter.

As the organic compounds are vaporized, they are immediately oxidized to carbon dioxide in an oxidizing oven immediately behind the sample oven. The flow of helium containing the CO₂ then goes to a methanator oven where it is converted to methane which is then detected by a flame ionization detector.

2. After cooling to 525°C, a 2% oxygen/helium mixture is switched into the sample oven. This is followed by temperature steps of 600°C and 700°C. During this phase, both the original elemental carbon and that produced by the pyrolysis of organics during the first phase are oxidized to carbon dioxide, followed by detection as described in part A. Again, the darkness of the filter is continuously monitored.
3. After all carbon has been oxidized from the sample, a known amount of methane is injected into the sample oven. This serves as an internal quantitating sample for each analysis and assures that the instrument is operating properly.

Based upon the FID response and laser-transmission data, the amounts of organic and elemental carbon are then calculated for the sample.

B. Sample Collection

Samples intended for this analysis must be collected on quartz fiber filters (e.g., Pallflex QAO-UP, Micro Filtration Systems QR100, etc.). The punch 1.0 cm by 1.5 cm is used to cut standard sized filter aliquants for analyses. Sufficient filter sample must be sent to allow at least one repeat analysis if necessary. In order to take advantage of the low detection limits, the filters must be pre-fired in air (700°C for about an h) in order to remove any residual carbon contamination.

At this time, we will analyze samples collected on glass-fiber filters. However, extra precautions must be taken to prevent the molten glass from coming into contact with the internal quartz oven. Also, because the temperature of the final step with helium must be lowered to prevent the glass from melting during the period while the absorbance is being measured, there might be a small error which may slightly overestimate elemental carbon and underestimate organic carbon. Please check with us before sending samples in glass.

The best range for the deposit concentration is 5 to 200 micrograms per square cm for organic carbon and 1 to 15 micrograms per square cm for elemental carbon. Concentrations of organic carbon greater than this have a possibility of going off scale, as well as possibly interfering with the elemental carbon measurement when only small amounts of elemental carbon are present. Values of elemental carbon greater than about 15 micrograms per square cm tend to be too dark which, as with any optical absorbance method, makes results hard to interpret.

C. Precision, Accuracy and Detection Limits

The precision of this analysis is nearly always in the 4-6% range for samples collected with the above specified ranges. This applies to the organic and elemental carbon speciation values as well as total carbon. For sample sets greater than 10, duplicated analyses are usually made on 10-15% of the samples and reported to give a measure of precision. Note that this value also incorporates any variation in the sample density on the surface of the filter.

Accuracy of this method for total carbon can be determined by analyzing a known amount of carbon. This is done periodically as part of our quality-assurance program by the addition of a known amount of sucrose or lactose to a blank filter, which is then analyzed with the normal procedure. Results again always tend to agree with predicted values within the 4-6% range.

Accuracy of this method to determine the speciation of organic and elemental carbon is not so easily measured, since it is, as stated before, a method-dependent parameter. Several experiments have been performed, however, to create mixtures of known amounts of organic and elemental carbon which

act as real-world samples during the analysis by pyrolyzing some of the organic carbon. These experiments have always given the predicted values for the speciation within 5-10%, depending upon the relative amounts of organic and elemental carbon on the filters. Several method intercomparison studies have also been done and the results have been very comparable.

The detection limit of this method is on the order of 0.2 to 0.3 micrograms per square cm of filter for both organic and elemental carbon. For this reason, the filters must be pre-fired as described above. New quartz fiber filters which have not been fired have been found to have from 2 to 10 micrograms of organic carbon per square cm.

D. Analysis Interferences

The majority of aerosol samples collected using a size-segregation inlet (less than 10 microns) have been free of interference problems. However, there are some instances where problems might occur.

Certain places where samples are collected might contain inorganic carbonate particulate matter, such as might occur from some types of dust or certain types of industries. This is usually eliminated by a size-segregation inlet, but some may still end up on the filter. An absolute upper value for this type of carbon can be given, since the temperature steps during the first phase of the analysis were selected so any inorganic carbonate would appear in the fourth peak of the analysis.

The presence of colored inorganic compounds, the most common being iron oxide, does not usually cause a problem as long as there is not a heavy deposit. This is because the absorbance of the He-Ne laser light is usually small compared to the absorbance of any elemental carbon present and because the absorbance almost always remains constant during the analysis. The only time it has been noted that colored inorganics have been a problem is where there is some type of local source, such as copper smelters or other ore-producing industries, which produces a compound which can change color such as forming a black oxide during the oxygen phase of the analysis. If you suspect such a possibility, please check with us first.

Colored organic materials, however, are somewhat common, especially in areas impacted by the burning of biogenic material. Again, the fact that these materials have an absorbance coefficient for the He-Ne laser light which is small compared to the absorbance of elemental carbon makes the error relatively small. In instances where the amount of elemental carbon is relatively very small and there is a large amount of absorbing organics, it has been found that 1 to 2% of the organic carbon can be mis-assigned to the elemental carbon fraction.

It has been determined that the presence of non-absorbing inorganics, such as ammonium sulfate or nitrate, which scatter light and which vaporize during the analysis, have only a small negligible effect on the determination of elemental carbon. Only if the levels of such inorganics were very high ($>100 \mu/\text{cm}^2$) and the amount of elemental carbon very low is there much error.

E. Results and Turn-Around Time

Sample sets containing less than about 25 samples can usually be analyzed and reported within two to three weeks. Sets larger than 25 may take longer depending upon current work load. These larger sets can be scheduled, however, so that results can be obtained quickly. Typically, about 70 analyses per week are analyzed.

Results are reported as micrograms per square centimeter of the filter deposit of carbon in the organic fraction and in the elemental fraction, as well as inorganic carbonate if present. If the necessary data are provided on a commonly used spreadsheet format (either MS-DOS or Macintosh) the resulting data can be inserted for calculations of results such as micrograms per cubic meter of sampled air. A hard copy printout will be sent by mail. In addition, results can also be obtained as a data file on floppy disk for most of the commonly used spreadsheets (e.g., Lotus 123, SuperCalc 5, Excel-Mac).

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Standard Procedure for the Micro-determination of Carbon and Hydrogen

A. Preparation for Combustion

1. Solid (non-volatile) samples
 - a. If requested, sample is dried as directed.
 - b. The work surface is wiped with a non-linting paper tissue. The spatula is carefully cleaned with a non-linting paper tissue and visually inspected for cleanliness.
 - c. A clean aluminum boat is placed on the balance and the balance is zeroed.
 - d. The appropriate amount of sample to be analyzed is transferred to the boat and the determined mass and sample number are recorded.
 - e. The boat and contents are removed from the weighing chamber of the balance and placed on the catalyst stand and the boat is packed with catalyst. The boat and contents are placed in micro-desiccator or combustion ladle as appropriate.
2. Volatile (solid or liquid) samples
 - a. The work surface is cleaned with non-linting tissue.
 - b. A new glass pipet is flamed. (Under no circumstances is a pipet reused.) The used pipet is discarded after use.
 - c. A flamed glass sample holder is placed on the balance and zeroed.
 - d. The sample is transferred to the sample holder. Using the micro-flame, the sample holder is sealed. The mass and sample number are recorded.

- e. Remove sample holder to catalyst stand and place in the micro-desiccator or combustion ladle as appropriate.

B. Sample Combustion

1. An MHW Laboratories modified pregl-type combustion apparatus is used to combust samples.
2. The following systems apparatus make up the combustion
 - a. Combustion furnace
 - b. Internal tubing
 - c. Thermal conductivity cell
3. The combustion ladle is cycled to insure there is no significant organic material carried into the combustion zone upon injection.
4. The modified carbon-hydrogen combustion apparatus must be calibrated with standard compound which contain known amounts of each element.
5. The combustion ladle with standard sample is loaded into the combustion port and the combustion tube is purged of admitted air.
6. When the admitted air has been purged and the combustion temperature has recovered to between 1025 and 1035°C, the ladle is injected into the combustion zone for the period of time necessary to insure complete conversion of all carbon in the sample to carbon dioxide and all the hydrogen in the sample to water.
7. Blank Determination
 - a. Three blank determinations are performed by each analyst. The magnitudes of the blanks are evaluated for consistency. (The absolute values of the 'blanks' are not important, but are recorded.)

C. Measurement

1. The resulting carbon dioxide and water are measured by a thermal conductivity detector (TCD).
2. The output of the TCD is directed to the Integrator (H-P 3394A) and percent carbon and hydrogen are recorded.

D. Standard Compounds

1. Three different standard compounds are combusted and are results are evaluated to ensure proper combustion.
2. A standard compound is combusted periodically during an analytical run to assure the apparatus response is constant.
3. Submitted samples are not considered as standard compounds.

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Standard Modified Dumas Procedure for the Micro-determination of Nitrogen

The method for nitrogen analysis is an MHW modified Dumas procedure. The combustion gases are separated chromatographically using helium as the carrier gas. The resulting nitrogen is measured by a thermal conductivity detector.

A. Preparation for Combustion

1. If requested, sample is dried according to instructions.
2. Mass is determined (Cahn Model 4700 Electro-Balance) by the procedure as outlined in LOI-001 Section A.

B. Sample Combustion

1. MHW modified Dumas-type combustion apparatus is used.
2. The apparatus is allowed to warm up prior to analysis to ensure stable and consistent response.
 - a. Oxidation temperature is 1000°C
 - b. Reduction temperature is 550-600°C
 - c. Flow is 120 mL/min⁻¹
 - d. RT time is 1.20 min.
 - e. TCD temperature is 94°C

C. Measurement

The procedure outlined in LOI-001 Section C is followed.

D. Calibration

1. Three different standard samples are combusted at a temperature in excess of 1000 Centigrade for the standard time. The results are evaluated to ensure linearity, *i.e.* all values are within $\pm 0.3\%$ of the theoretical values.

2. Calibration standards are:

- a. Acetanilide (N.B.S. standard or equivalent) %N=10.36
- b. 2,4 DNP (N.B.S. standard or equivalent) %N=20.14
- c. Urea (A.C.S reagent) %N=46.64
- d. Melamine (organic analytical standard) %N=66.64

E. Blank Determination

Three blanks are combusted prior to sample analysis and results are evaluated to ensure that no nitrogen is found.

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Cahn Balance Calibration Procedure

Cahn Instruction Manual #4930 is the basis for this LOI. The manual should be consulted for complete information concerning all aspects of the care, service and operation of the Model 4700 balance. The following is not meant to be definitive.

A. Set up

1. The balance is set up according to the manufacture's instructions.
2. Balance is left on at all times to avoid inordinate so that warm-up time.

2. Calibration

1. A clean aluminum boat is placed on the left stirrup and the counterweight (tare 9) is observed to be secure on the right stirrup. The Balance is zeroed.
2. The 2 mg class "M" precision weight is placed carefully in the boat on the left stirrup.
3. The digital volt meter (DVM) is set to 2.000 ± 0.0001 . The DVM is allowed to stabilize for at least 30 s. (3.5)
4. The balance calibration is checked at the beginning of each work day.

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APPENDIX J. LETTER OF INSTRUCTION

OREGON GRADUATE INSTITUTE OF SCIENCE and TECHNOLOGY

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R.A. Rasmussen
September 18, 1995

Letter of Instruction

OB/OD DUGWAY, UTAH

Quality Assurance Objectives

Gas Samples

Whole air samples will be collected in evacuated stainless steel (SS) internally electropolished SUMMA® canisters in accordance with the EPA Compendium TO-14 method. This method is the most appropriate approach for performing the required tasks of: providing certified clean 6-L SUMMA® canisters; determining the TO-12 TNMOC; hydrocarbon speciation via the TO-14 method; analyses of the fixed gases CH₄, CO, CH₂; GC-MS full scan measurements of the organics from peaks in the TIC greater than 25% of scale; and special SF₆ tracer analyses.

Design and Fabrication of TO-14 Sampling System

OGI will provide 2 Metal Bellows (Model 158) pumps to collect grab air samples at a sampling rate of 28 L/min. These pumps will fill the 6-L cans from -30 mmHg to +20 psig in < 3 minutes. Annealed PFA-type of Teflon® ¼-inch lines will be used to collect the air samples. These sampling lines have been specifically designed and fabricated for the needs of the VOCs in the OB/OD study and connect directly at Ports 1 and 2 of the Bang Box manifold. The pumps have been successfully used in many air pollution studies and in earlier Bang Box tests. They are individually certified to be ultra clean by the EPA TO-12 and/or TO-14 methods before use in the field. Each canister to be filled from the pump is connected by a purge-tee to provide immediate dynamic flushing of the line before the valve on the canister is opened. All of the pumps will work off 110 VAC. The pumps will be tested manually by the technician collecting the samples before the test detonations. Tables 1 and 2 are examples of the chain of custody and field sampling sheets. OGI will install and operate the sampling systems at the Dugway (Bang-Box) site. Each sample collected is documented as to site, date, time, canister size, and start and final pressures. The whole air collection system sampling line intake point just outside the vestibule door in the Bang Box is fabricated of internally electropolished 1-inch O.D. SS tubing, including the manifold. The TO-14 sampling procedure installed at Dugway has been used successfully in the previous

OB/OD studies.

The multiple samples to be collected for SF₆ will be done using 0.85L SS canisters in conjunction with both the whole air sampling port and the special PFA Teflon® lines positioned at other points within the Bang Box.

Note that every canister is individually certified to be clean (< 10 ppbC) before it is sent out to be used. In Figure 1 is a frequency distribution of 133 SS SUMMA® canisters "certified clean" by the EPA TO-12 method and then retested with moist zero air after being held at 155°C for 24 hours. Any residual organics on the walls would have desorbed or degraded at this temperature. These cans are especially clean at 1-3 µg/m³ (1 µg/m³ = 1.67 ppbC).

Sampling and Analytical Procedures

Volatile Organic Carbon (VOC)

OGI has pioneered over the past 20+ years the GC-FID and GC-MSD analyses for full speciated measurements of VOCs in the atmosphere in both urban and rural situations using the "SUMMA®" SS canisters Professor Rasmussen invented in the 1960s for VOC sampling. In Exhibit 1 is a list of 610 compounds measured by GC-MS-Full Scan to 0.1 ppbv in our laboratory using the TO-14 method from air collected and stored in SUMMA® canisters. This is the most extensive list that exists to date for C₃-C₁₅ analytes recovered from SUMMA® canisters. This list will be the search list used in the analysis required in the OB/OD project. The list includes many compounds that contain chlorine, nitrogen, sulfur, and oxygen.

In Table 3 is the TO-14 air toxics list of 42 compounds that have been certified by EPA for SUMMA® canisters. Our precision of analysis for the TO-14 list is included in the table and a labeled GC-MSD TIC-chromatogram is shown in Figure 2 for the TO-14 list.

The precision of analysis reported for our TO-14 measurements is generally less than 5%. This is typically less than what EPA requires. We have found that our GC-MS systems are more stable than what is generally associated with this type of instrument performance because our GC-MSs are dedicated to solely analyzing air samples with analyte loadings controlled for 1-5 µg. Optimizing on this narrow range of mass loadings has greatly improved day-to-day performance.

Our GC-MSD operating specifications are given in Tables 4 and 4A. These operating conditions are routinely used in two modes. Loading 4 μg of analyte onto the head of the column is used for full scan TIC sensitivities of 10-20 pptv for individual peaks. The method is semi-quantitative because peaks with large concentrations, i.e., benzene, will saturate the detector and give lower than correct values. However, the method is used to survey for very low levels of toxic analytes re identification of every compound in the TIC chromatogram. Lower loadings, < 1 μg , of analyte are used for better quantification of the major peaks in the TIC chromatograms, especially benzene, toluene, and other species that do not have many minor ions to measure.

The GC-FID hydrocarbon VOC conditions of analysis are given in Table 5. This method very accurately quantifies the hydrocarbon $\text{C}_2\text{-C}_{10}$ ozone precursors (70 compounds) listed in Table 6. (Note: the EPA PAMS list requires 55 hydrocarbons be speciated in order to be used to estimate the ozone-hydrocarbon potential of an air mass.) A summary of the analytical and quality assurance specifications for measuring VOCs, air toxics, and related trace gases in the OB/OD air samples is given in Table 7. The GC-FID analyses are used primarily for careful quantification of the hydrocarbon species compared to semi-quantitative measurements by GC-MS and special low to sub parts-per-trillion analyses for exotic fluorocarbons (Table 4A).

The determination of total nonmethane organic compounds (TNMOC) in ambient air samples using cryogenic pre-concentration and direct flame ionization detection (PD-FID) is made using the EPA Compendium Method TO-12. The GC-FID method's conditions of measurement are given in Table 8. A diagram of the operation of the method followed is shown in Figure 3. The calibration against NIST SRM propane is shown in Figure 4. An example of the TO-12 analysis of a real sample is shown in Figure 5. The multiple components suggested in the OB/OD TO-12 measurement is characteristic of complex samples. This information is necessary to optimize subsequent GC-MS, FID, ECD, etc., analyses.

The SF_6 analyses are used to determine the volume of the air-supported Bang Box building, homogeneity of mixing immediately after the detonations, and the rate of dilution as fresh air is ducted into the building. The SF_6 measurements are made by a special GC-ECD method. The specific conditions of analysis are given in Table 9.

The fixed gases $\text{CH}_4\text{-CO-CO}_2$ are measured on both the whole air samples (6-L cans) used for VOC characterization and on the (0.85-L) cans obtained for the SF_6 studies. The conditions of analysis are given in Table 10.

In Summary

The analytical work and quality assurance activities are directly under the supervision of Professor R.A. Rasmussen. The procedures followed in our laboratory to ensure data reliability from sample collection through final data reporting are:

1. The sampling pumps are tested in the field to ensure that they are being properly maintained.
2. The canisters are individually certified clean before shipment to the field. Travel blank canisters are tested with each field test site.
3. The canisters are attached to the sampler via purge-tee leak-tight Swagelok® fittings.
4. The sampler SS lines are flushed with ambient air via the sampling system pump being turned on and purge-tee vented during non-sampling portions of the operations. This eliminates any possibility of high levels of contaminants being trapped in the system lines between detonations or burns and drawn into the exposed canisters.
5. When the canister valve is opened, the vacuum is checked. If -29 to -30 inches Hg vacuum is observed, the canister is good. If not -28 or higher, then the canister is replaced with a new canister.
6. At end of the fill cycle, the canister final pressure is recorded. It should be 20 psig. Any operating problems are noted on the sample record sheet.
7. The canisters are returned via Air Freight to PDX and picked up by Mr. R. Dalluge or Mr. D. Stearns.
8. Canister date of receipt at OGI laboratory is noted and pressure measured at laboratory before analysis.
9. The canisters are equilibrated in our oven room (warm room) for 24 hours at 85°F before the canister is forwarded to the TO-12 and TO-14 (GC-FID/MSD) stations for analysis.
10. GC-FID TO-12 is calibrated daily against NIST SRM propane.
11. GC-FID TO-14 calibration is made daily against neohexane and NIST SRM standards. Results of SRM calibrations, FID response factor, and a system blank kept in instrument log book.
12. Precision of analysis is determined weekly from replicate SRM and CRM calibration analyses.
13. GC-MS full scan analyses are run primarily for qualitative identifications and/or semi-quantitative measurements for compounds not on the EPA TO-14 list.

- Autotune and recalibration against TO-14 standard are made biweekly or before special quantitative analyses are performed.
14. At least one EPA Quality Assurance audit is expected to be administered by EPA if so requested.
 15. Optimum sample loadings of < 1.0 to $5 \mu\text{g}$ of analyte determines the volume of the sample to be processed for analyses, i.e., an ambient TNMOC of $500 \mu\text{g}/\text{m}^3$ requires 3.0 Liters of air for $1.5 \mu\text{g}$ loading of the analytes for MSD analysis. The TO-12 TNMOC values are used for determining the volume of sample required for analysis.
 16. By analyzing only sample loadings that are within a narrow range, optimum analytical accuracy and linearity are maintained and obtained from the GC-MSD system. The GC-FID optimum VOC analyte/volume relationship is not as critical as the MSD, but 200 and 500 mL sample volumes are used for best results.
 17. Compound identifications are made using the NIST/EPA/MSDC Mass Spectral Database, Version 2.0 (DOS-based), the NIST (NBS) data base mass spectral library PBM format in conjunction with the HP59970 MS Chemstation® (Pascal) operating software for the MSD and confirmation of high resolution GC-FID VOC analyses.
 18. The data reduction reports are in Lotus 1-2-3/Windows prepared in whatever format is required, i.e., compound, data of analysis, major mass measured, CAS #, concentration of analyte, etc. Both hard copy (paper) and electronic data files on floppies are prepared for the report.
 19. All of the raw data in the computer files from the Chemstation® software program are saved on magnetic tape. This provides for extracting more detailed information from the FID or TIC chromatograms at a future date if necessary.
 20. A complete record of traceability from canister preparation, certification of cleanliness for both samplers and canisters, chain of custody of handling and analysis is maintained as well as EPA audits of TO-14 performance and calibration standards. These records are maintained for three years.
 21. Report of turnaround time is usually 14 days or less from the date of receipt of the samples.
 22. All documentation for OB/OD tests is stored in Mr. R. Dalluge's office area. This is a secure area.

Calibration Procedures

Volatile Organic Carbon (VOC)

Calibrations are made directly against NIST-NBS Standard Reference Materials (SRMs) for both: speciated hydrocarbons calibrated against propane, #1665B, or benzene, #1805, air toxics against SRM 1804 or EPA TO-14 CRM standards, and CH_4 , CO, and CO_2 against NIST SRMs. In addition, entries in the instrument log books for FID response factor (Figure 6) and MSD autotune voltage readings are kept. A very important part of our calibration procedures to obtain the highest quality measurement of ambient air samples by GC-MSD is to always measure the TNMOC in the samples first by the EPA TO-12 method. This enables us to then load onto the head of the column the optimum amount of analyte for the best GC-MSD analyses. This special part of our analysis protocol is especially relevant to the high variability of VOCs in the samples obtained in the different materials being studied in the OB/OD test facility at Dugway.

The results from long-term periodic audits of our daily calibration for benzene are shown in Figure 7, a 9-year record. The results from an EPA audit for a DOD project requiring special attention for low levels of benzene measured in ambient air samples are given in Table 11. Recently we finished an audit for the TO-14 analytes requested by Region X (Seattle) per an EPA project in Fairbanks, AK. This audit is being administered by Dr. Bill Mitchell, Head, Quality Assurance Division, EPA, Research Triangle Park, NC. Our last OB/OD audit by EPA on August 24, 1993, was an on-site visit by Mr. Curtis M. Morris. His report is attached as Table 12a, b, c.

APPENDIX K. LETTERS OF INSTRUCTION

RADIAN CORPORATION

Title	LOI
Quality Assurance/Quality Control: Procedures for Accuracy, Precision, and Completeness . .	10
SVOC Sampling Procedure	26
XAD-2®Preparation	27
Sample Preparation	28
Gas Chromatography/Mass Spectrometry (GC/MS)	29
Sample Custody and Identification	32
Dioxin and Furan Sampling and Analysis	33
Determination of Hydrogen Chloride Sampling Procedure	39

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**QUALITY ASSURANCE/QUALITY CONTROL:
PROCEDURES FOR ACCURACY, PRECISION, AND COMPLETENESS**

1) QUALITY ASSURANCE (QA) PROCEDURES

QA Procedures include the following:

- Procedures to be written, peer reviewed for technical soundness, approved, and followed;
- Project personnel to receive or have adequate training to conduct the necessary tests to meet work assignment objectives;
- Work assignment activities to be documented in a manner that will not compromise data integrity and will facilitate reconstruction of the work;
- Data management to include procedures for data filing, storage, and retention to safeguard data integrity and security;
- Internal quality control activities to provide ongoing verification of the technical soundness of the work and accuracy of the data; and
- QA activities to be conducted by personnel not involved in method development to provide an ongoing objective assessment of the effectiveness of the internal quality control program.

Additional procedures used to maintain the quality and usefulness of the data generated during this work are frequent meetings (daily/weekly) with the project staff to resolve any technical problems, assess the progress, and discuss technical results.

Field studies are quantitative and thus require a quality assurance program which allows the production and statistical evaluation of data of sufficient quality to meet program objectives. This narrative LOI is a description of the QA items developed to help ensure that the evaluation process is well planned, adequately documented, reproducible, and auditable.

INTENDED USE OF THE DATA

The data from this effort will be used to determine emission factors from materials during a 10-Item Test program and provide scientific data to answer the question of environmental acceptability of OB/OD thermal treatment methods. Data from the various members of the sampling and analysis teams will be collected and slated for submission to the Dugway staff.

DATA QUALITY OBJECTIVES

Data quality objectives are determined by assessing previous measurement data and method evaluation studies. The interim data quality objectives include requirements for method precision, accuracy, and completeness. The procedures for determining precision and accuracy are described in the definitions provided at the end of this section.

Data quality objectives for completeness were established to ensure that the individual methods are sufficiently reliable to generate valid data with a minimum number of attempts. Laboratory method evaluation studies use completeness as a criterion and compare actual attempts to the actual number of data points collected. Appropriate procedures are implemented to help curtail sample loss to ensure that the data completeness objectives are met for each analytical phase. These special QA procedures could include modification of sample preparation procedures, sample handling, storage, and analytical procedures.

Sample representativeness will be assured by performing the appropriate number of sampling and analytical runs with sufficient numbers of sampling devices. These experimental factors are discussed in detail in the individual Test Design Plan sections regarding test items and sampling matrix.

Standard procedures for the preparation and analysis of the samples will be followed to ensure comparability. If deviations from these procedures are required or inadvertently occur, descriptions of these deviations will be recorded in the project notebook or on data worksheets. Prior to reducing the data, the impact of the deviations from standard procedures on the results will be discussed with the appropriate Project Manager/Task Leader and documented in a note to the file. The data will also be flagged to show that they may not be comparable.

4) DATA QUALITY: DEFINITIONS

The terms used to define data quality are as follows:

- **Precision** is an estimation of the deviation of the results from the analyses of identical samples or samples collected at the same point in time and space. The precision calculated from these measurements also includes random errors due to sampling and analysis. Analytical precision can be determined from laboratory replicate samples. This estimation of precision is more clearly defined as repeatability. When the results are near the detection limit, matrix spike/matrix spike duplicates can be prepared to assess the recovery of the target compounds and the precision of the results. The spikes are prepared at measurable levels. Precision is usually defined in terms of standard deviation, relative standard deviation, or coefficient of variation. Typically, precision results will be reported as percent coefficient of variation.

$$\%CV = S_x/X_b * 100$$

is where S_x is the standard deviation of x number of data points, and X_b the mean or average of x number of data points from the data set.

- **Bias** is the closeness of measurements to a reference value and is similar to precision in that the entire system or just the laboratory can be assessed. Where spiking is appropriate, samples are spiked to allow an assessment of as much of the measurement system as possible. When the entire system is assessed for bias, samples are spiked as collected. When determining laboratory bias, a representative sample is divided, spiked, and the recovery results used to estimate bias. Spiking concentrations are above the expected target analyte concentrations. The percent recovery is calculated as follows:

$$\%R = [\text{Result/Total Spiked}] * 100$$

Bias is expressed as a difference or percent difference (%RPD) between measured and known values.

$$\text{Bias} = X - T$$

$$\%RPD = [X-T]/T * 100$$

where T is the true value (reference material) and X is the mean sample concentration. The relative percent difference is indicated as %RPD. Matrix effects and analytical interferences will be considered when developing and analyzing reference samples. Bias will be tested for statistical significance.

- Completeness is a measure of the amount of valid data obtained from the measurement system compared to the amount of data planned. Completeness is based on previous experience following similar sampling, sample preparation, and analytical procedures. The level of completeness is often affected by analytical and sampling difficulties which inherently affect the number of valid samples collected. Therefore, precaution is taken to avoid sample loss.
- Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, variations of a parameter at a sampling point, or an environmental condition. Representativeness is assured by collecting or preparing samples that are similar in every way possible and assuring that these samples will include all of the characteristics or analytes in the sampling environment that are required to be assessed. The sampling procedure is often developed by a statistician and is dependent upon the sample. The sampling procedure could also be determined from historical data.
- Comparability is a measure of the confidence with which one data set can be compared to another. The comparability of the data sets also required compliance to standard procedures and use of the same data quality indicators. If the data quality indicators, collection procedures, measurements, and methods are equivalent, the data sets can be compared.

All field and laboratory personnel will be familiar with the project objectives for quality and the standard procedures for conducting this work assignment. Periodic reviews of the work will be conducted by the Project Manager. These progress reviews include evaluations of the implementation of standard procedures and achievement of the data quality objectives. If problems are found, actions are taken immediately to bring the system under control. A reassessment of the applicability of the standard procedures and reasonableness of the data quality objectives could be required if problems are encountered.

5) DATA DOCUMENTATION, REDUCTION, VALIDATION

Because data quality is related to good documentation procedures, project activities are documented in bound laboratory notebooks or on worksheets in a manner that allows reconstruction of the work and verification of the conclusions drawn. Data entries are in black ink and corrections are made so as not to obscure original entries.

These procedures will be followed during this effort and the documentation of results will be adequate to reconstruct all activities. Where applicable, worksheets will be developed and used to record results from instrument readings and data manipulations. All deviations from the approved procedures will be recorded in the project notebook and reported to project management to allow a complete assessment of the overall impact on the project.

Data reduction is the responsibility of the person generating the data. Where large volumes of data are generated, this responsibility may be delegated. However, the data generator is ultimately responsible for data accuracy. Data reduction procedures will be developed and described in the project notebook. A statistician will be assigned to the project to conduct any complex data evaluation activities. The statistical analysis of the data will also be described in the project notebook, approved by the project management, and summarized in the final report.

The data will be peer reviewed at sufficient intervals to verify the soundness of the technical approach, accuracy of the results generated, and adherence to the project plan. These reviews will be evidenced by summarizing the findings and actions taken in response to recommendations for corrective actions in a note to the data file and Project Manager. Data reviews are also evidenced by initialing and dating validated pages of the project notebook and work sheets.

6) QUALITY CONTROL

The quality control (QC) procedures for this effort are based on quality requirements defined in Radian's corporate quality program and site specific standard operating procedures (SOPs). The list of documents used to develop these procedures and requirements includes the following:

- U. S. EPA "AEERL Quality Assurance Procedures Manual for Contractors and Financial Assistance Recipients";
- EPA-600/9-76-005, "Quality Assurance Handbook for Air Pollution Measurement Systems";
- 21 CFR 58, Food and Drug Administration "Good Laboratory Practice for Nonclinical Laboratory Studies";

- 40 CFR 792, EPA "Good Laboratory Practice Standards"; and
- ANSI/ASME NQA-1, "Quality Assurance Program Requirements for Nuclear Facilities."

The quality control procedures most applicable to this effort are:

- Conduct of the work by adequately trained personnel;
- Supervision of the work to ensure that the work plan and QA/QC procedures are followed;
- Documentation of experimental results in project dedicated notebooks in a manner that will facilitate reconstruction of project activities and verification of data accuracy;
- Review of the progress of the work by senior technical reviewers and independent QA personnel to assess the effectiveness of the internal quality control program;
- Use of traceable standards and accurately prepared test substances;
- Use of equipment that is calibrated and tested according to laboratory standard operating procedures;
- Documenting routine and non-routine maintenance of equipment to allow an assessment of reliability;
- Following sample and reagent labeling procedures to help prevent sample mix-ups;
- Analyzing reagent and matrix blanks to assess sample and analytical system contamination; and
- Testing QC samples to assess the appropriateness of the analytical system and sample preparation reagents.

These quality control activities are described in greater detail in the individual letters of instruction and laboratory standard operating procedures. These procedures have been developed and implemented to help ensure that the data produced by the analysts are of sufficient quality to meet the data quality objectives. The effectiveness of the quality control program is evaluated during internal project

reviews conducted by project management and external audits performed by QA personnel.

7) QUALITY ASSURANCE

The quality assurance program for this effort includes the conduct of systems and performance audits to determine if the quality control procedures are adequate to provide defensible data of known quality. Performance audits are conducted to determine if the approved procedures are followed and performance criteria met. Systems audits involve assessments of the effectiveness of the quality control and managerial systems established for the work. These audits are conducted by QA personnel who are not actively involved in the conduct of the work.

The quality issues assessed during the audits could include several of all of the following:

- Project management;
- Employee training;
- Staffing;
- Adherence to letters of instruction and standard operating procedures;
- Sample custody;
- Calibration and quality control procedures;
- Equipment operation and documentation of maintenance;
- Data reduction/validation; and
- Record keeping.

Audit findings are discussed with the project personnel and project management immediately after the audit. Audit findings are also summarized in a report distributed to management for immediate attention and resolution of any problems or recommendations made to improve the quality control program.

2) CORRECTIVE ACTION MECHANISMS

In order to ensure that quality concerns and problems are eliminated, corrective actions are formally documented and the actions taken to correct them monitored. Several mechanisms for ensuring immediate resolution of these problems are used by Radian.

Recommendations for corrective actions (RCAs) are issued by QA personnel when systematic problems are found during audits. The actions taken in response to these recommendations are verified by the auditor prior to closing the audit file. A computer tracking system is used to track RCA issuance, identify the person responsible for problem resolution, and monitor the verification of actions taken.

Problems identified by project personnel which pose a serious threat to data quality and the achievement of project objectives are documented on Corrective Action Request Forms (CARs). These problems are immediately brought to the attention of project management and resolved as soon as proposed corrective actions are approved. The completed CARs are retained in the project file.

3) REPORTING

The reports generated from this effort will be developed and forwarded to the Project Manager. The reports are reviewed by the other technical reviewers prior to submission to the Project Officer. These reviews include assessments of the accuracy of the results reported, clarity of the information presented, and technical soundness of the conclusions.

The report and any preliminary reports will include at least the following:

- Project number; Radian Document Control Number (DCN);
- Dated title page; Table of contents;
- Objective of work and whether it was achieved;
- Procedures followed and whether they were executed as indicated in the test design plan;
- Data quality objectives and whether they were achieved;
- Tabulation of results; Quality control results; and
- Summary of findings.

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Tables will be numbered and the units of measure given. Data manipulations will be clearly explained along with any statistical analyses of the data. References to other documents will include sufficient information to facilitate their retrieval.

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SVOC SAMPLING PROCEDURE

A. Equipment Description

The field monitoring for SVOC's will be performed using General Metal Works, Inc. (GMW) Model PS-1 air samplers. XAD-2 resin will be used to collect the SVOC samples from the ambient air.

1. PS-1 Sampler

The sampling system is housed in a standard high volume shelter that contains: a special sampling inlet (i.e., aluminum sampling module), a venturi-Magnehelic gauge assembly, seven-day Paragon timer, elapsed time meter, voltage controller, ball-valve, motor, and exhaust hose. Power requirements are 115 volts AC at 60 hertz drawing 8.0 amperes. A schematic of the PS-1 sampler and its components is presented in Figure 1.

2. Aluminum Sampling Module

The aluminum sampling module is designed to hold a 100 mm diameter quartz fiber filter (Whatman QMA) at its inlet and a glass sampling cartridge in its lower canister. The sampling cartridge is a 60 mm i.d. (63.5 mm o.d.) x 125 mm long borsilicate glass cylinder. A narrow indentation encircles the cylinder approximately 25 mm from one end and holds a coarse glass frit support.

3. Operation of PS-1 Sampler

The PS-1 Sampler is operated by moving the switch at the lower part of the seven day timer to the ON position. Air flow through the sampler is measured by an in-line venturi-Magnehelic gauge assembly located immediately downstream of the sampling module. The sampling flow rate is set by adjusting the voltage controller connected to the pump motor until the Magnehelic gauge is at the desired value.

During this program, the samplers will be started and stopped manually. An elapsed time meter, connected between the timer and the pump will be used to verify the elapsed run time.

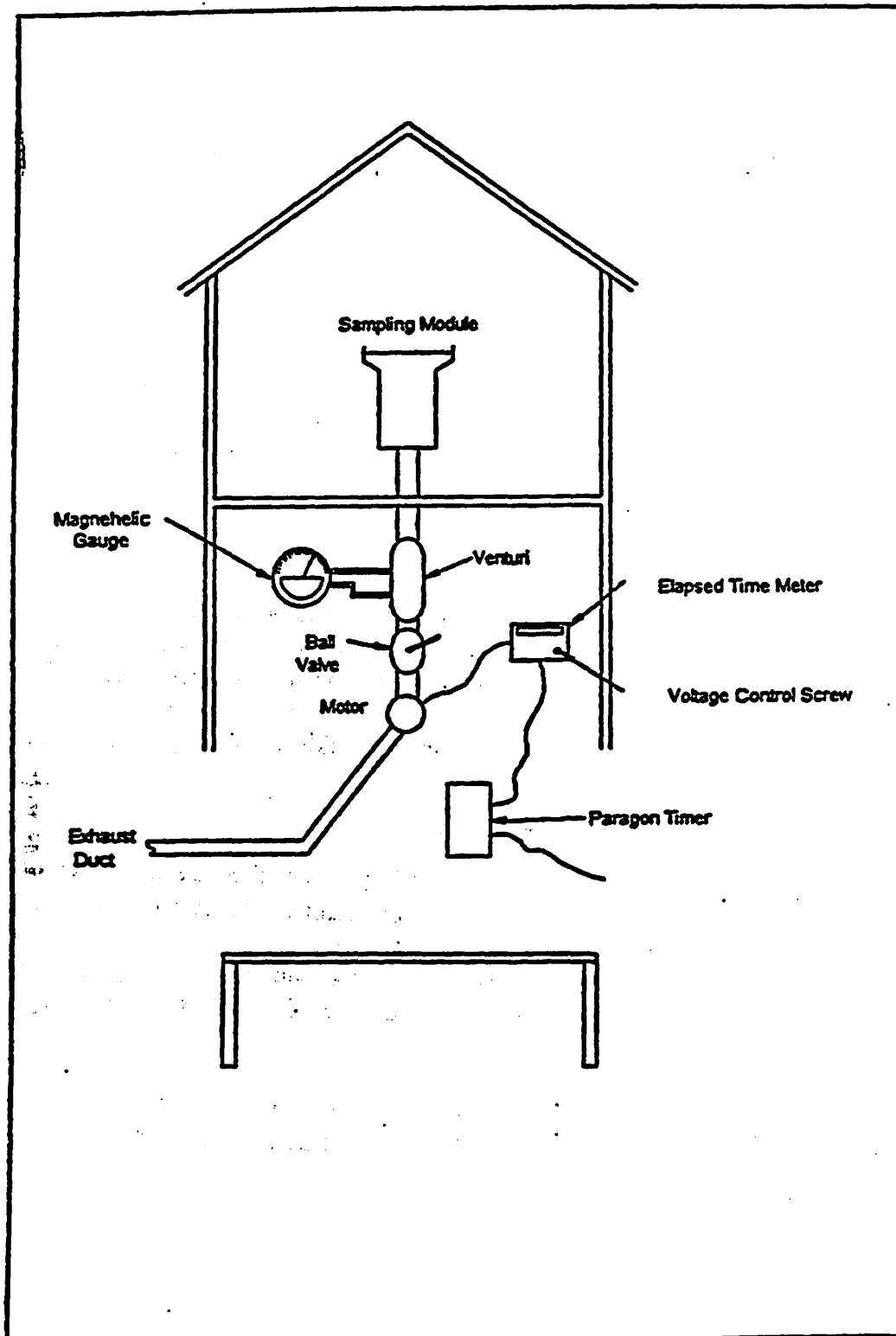


Figure 1- Schematic of PS-1 Sampler

4. Maintenance of Sampling Modules

Prior to, and following each sampling episode, the sampling modules should be thoroughly washed with soap (alconox or equivalent) and water and rinsed with distilled water. After drying, each module is wrapped in aluminum foil and placed in Ziploc bags for dust protection.

B. Media Preparation and Recovery

1. Preparation of the Sampling Module

For the SVOC sampling, a borosilicate glass cartridge measuring 60 mm i.d. by 125 mm long, will be loaded with XAD-2 resin to a depth of approximately 2.5 inches (approximately 45 grams of resin). The standard PS-1 sampling cartridge will be modified with a coarse quartz frit replacing the standard screen.

Prior to the preparation of the resin cartridge, the glass cartridges will be cleaned by thoroughly washing each cartridge with soap and water. After baking the glass cartridge, it will be rinsed with distilled water, hexane, and methylene chloride rinses, respectively.

Once the cartridge is cleaned, it will be wrapped in hexane/methylene chloride-rinsed aluminum foil and placed inside a padded foam lined sample jar.

The XAD-2 resin that will be used to collect the SVOC's will be obtained from the manufacturer and recleaned according to LOI 27 (XAD-2 Sorbent Preparation). The XAD resin cartridges will be cleaned in the laboratory and transported to the field for final preparation and sampling. The filters for the PS-1 samplers will be precleaned (solvent extraction with dichloromethane), dried and transported to the field for use in sampling.

2. Loading the Organic Trap into the Sampling Module

NOTE: Always wear appropriate gloves (NDEX® nitrile gloves, polyester gloves, or equivalent) when handling the SVOC media to avoid contaminating the samples.

To preserve the XAD-2 and provide a more efficient means of transporting resin material to the field, the clean resin will be shipped in a large container (4 Liter bottle or equivalent) and poured into the sampling cartridges right before sampling. This bottle of "clean" resin can then be recapped and used to fill cartridges in succession as required.

Remove the glass trap from the jar or can and orientate it so that the indented ring in the glass is down. Unwrap the foil carefully so as not to rip it so that it may be used to rewrap the trap after sampling. Fold the foil so the surface that touches the trap goes inward. Fill the trap with the designated volume of XAD-2 resin by pouring carefully from the "clean" bottle of XAD-2 to a depth of approximately 2.5 inches.

Open the aluminum sampling head, holding it so the small opening faces down. Make sure that there is a gasket both above and below the sample cartridge. Using teflon coated tweezers, lift the glass cartridge into the sampling module. Screw the other half of the module back into place without over tightening which may cause the cartridge to crack.

3. Loading the Filter

Loosen the three screws and pull them down to the side. Remove the aluminum plate and ring. Remove the thicker of the teflon rings using the teflon coated tweezers. Place one 100 mm diameter quartz fiber filter (Whatman QMA), rough side up, on the second teflon ring on top of the metal screen. Replace the first teflon ring on the filter. Ensure that the filter is centered. Replace the aluminum ring and plate and retighten the retaining screws. Insert the sample module into the sampler. The sample module will be prepared one hour prior to the initiation of sampling.

4. Loading the Sampling Module in the PS-1

Open the PS-1 sampler hood and lock open. Remove the aluminum foil from the lower tip of the sampling module and insert the module above the orifice of the PS-1. Lock down the two rings on the side of the orifice to ensure a good seal with the rubber gasket. Loosen the three top screws on the sampling module and remove the aluminum plate so the quartz fiber filter is exposed. Place the plate and the foil in a Ziploc bag, then replace the screws and tighten.

5. Sampling Initiation

Note the initial timer reading, barometric pressure, ambient temperature and clock time on the data sheet. Verify that the ball-valve lever is in the open position (vertical). Move the power switch to the ON position, and adjust the voltage controller until the magnehelic gauge shows the proper setting. Record the reading on the venturi-Magnehelic gauge. Place the timer ON and OFF toggle switch in the ON position. Record the start time of the sampling run. Immediately evacuate the test chamber to the designated zone for detonation sequence. Record detonation time.

If remote start and/or remote readings of the magnehelic gauge are possible due to improvements in the sampling hardware, those specific times and readings will be recorded in the field notebooks.

6. Sample Recovery

Following sampling runs, check the barometric pressure, ambient temperature and the elapsed timer to make sure that the sampler ran for the expected time. Replace the aluminum plate and aluminum foil on the module and move the sample to the recovery area making sure to keep the module upright. (Polyester gloves should be worn whenever the samples are handled to prevent contamination). Open the original foil wrap for the filter. Remove the quartz fiber filter with the tweezers and place it particulate laden side up.

In the case of the 10-Item Test to be performed in the BangBox in the winter of 1995, three PS-1 samplers will be running concurrently collecting samples. A munitions test will generate three composite samples of filters and XAD-2 resin traps. For multiple tests of a single munition, the quartz fiber filters and XAD-2 traps are replaced in an individual PS-1 sampler after each detonation. This is accomplished by physically removing the sampling heads (filter and XAD-2) containing samples and replacing them with new heads containing clean sampling media. The sampling heads can then be taken to the sample media preparation area to be taken apart ("broken down") for shipping. The resins and filters for each trial will be combined and labeled appropriately for the analytical laboratory. The glass traps and filters are removed by trial and wrapped for shipment to the analytical laboratory as described in the following sentences.

Open the lower part of the module and remove the glass trap. Place the cleaned foil over the top (open end) of the trap. Wrap the filter(s) and foil around the glass trap and place it in the Ziploc bag. Affix a sample label to the baggie and the sample jar. Collate the samples so that a XAD-2 trap and filter are packed for each PS-1 sampler. Place sample back in the padded canister and seal jar with custody seal. Fill out a chain of custody for samples. The samples will be returned to the laboratories by overnight courier in the protection sample pack with blue ice. All SVOC samples will be shipped to Radian - PPK laboratory for subsequent analysis.

7. BangBox Blank

A blank of the bangbox test atmosphere will be collected. This sample will be handled exactly like an actual air sample. It is designed to detect residual or background compounds that might be picked up through handling the sample or exposure with the immediate environment and atmosphere. To prepare the field blank, assemble the sampling module as described above.

8. Trip Blank

A trip blank is prepared media that is not opened or handled in any manner once it has been prepared. It is used to assess any contamination that may have been picked-up during media preparation and field shipment.

C. PS-1

1. Sampler Calibration

Calibration of the three GMW PS-1 samplers will be performed using a GMW Model 40 orifice calibration unit (OCU). This OCU is initially calibrated by the manufacturer against a standard rootsmeter. The manufacturer's calibration is accepted only if a linear regression analysis of the data yields a correlation coefficient of at least 0.9998.

Each PS-1 sampler will be multi-point calibrated prior to, and at the conclusion of this program. This is done by placing the OCU on the sampler orifice and clamping it in. A tube is connected from the side port on the ODU to a U-tube manometer (0-20 inches in H₂O). Make sure the magnehelic gauge and the manometer are zeroed. Turn the voltage adjustment to its highest setting and turn on the sampler. Allow the sampler to run for ten minutes to warm up.

The flow calibrations will be done at settings of 5,10,15,20, and 25. The rate is lowered by closing the ball valve, not lowering the voltage adjustment. Record the barometric pressure, ambient temperature, the magnehelic setting, and the manometer reading. If the flow rate decreases more than 20 percent during an individual sampling run, flow will be determined using flow values obtained between the sampling starting and stopping times, using the multi-point calibration data for that day's runs. Figure 2 shows an example PS-1 calibration form.

2. Field Calculation

The data for calculating the sample volume will be recorded in a dedicated lab notebook. The barometric pressure will be measured at the on-site client laboratory with a mercury barometer periodically during each sample event. Ambient temperature in the BangBox will be verified with a hand held temperature readout device. Internal bangbox barometric pressure will be calculated from the ambient barometric pressure and positive test chamber pressure. All volume calculations will be performed with an automated spreadsheet at the conclusion of the sample run.

3. Sample Documentation

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All SVOC field samples will be documented using standard chain-of-custody forms. An individual sample code will be used to identify each sample, sampler, and munition trial.

GMW Model P-1 Calibration Form

Name: _____ Date: _____

Monitoring Location: _____

S-1 Shelter No.: _____ Barometric Pressure: _____

GMW Model 40 OCU No.: _____ Ambient Temperature: _____

Magnehelic
Gauge Reading

Manometer (ΔH)
Reading (in. H₂O)

OCU
Flow Rate (m³)

$$\text{Flow Rate (SCMM)} = [(\Delta H)^{1/2} \times C_f \times m + b]$$

$$F = (\text{Barometric Pressure/Temperature}) \times 17.95$$

$$n = 0.1046$$

$$s = 0.008$$

Figure 2. GMW PS-1 Calibration Form.

XAD-2® PREPARATION

This LOI describes the procedure for the preparation of XAD-2® for use as a sampling medium in PS-1 ambient samplers.

1. XAD-2® resin that is newly obtained from the manufacturer is soaked in water for three days (or longer). The water and fines are decanted off, and fresh water is added. The XAD-2® is soaked for another three days. The water and fines are again decanted, after which the new XAD-2® is cleaned in the same manner as previously used XAD-2®.
2. A 2L glass bottle is filled nearly full with XAD-2®. Enough methanol is transferred to the bottle to just cover the XAD-2®, and the resin is allowed to soak for 3 days. The methanol and fines are decanted. The bottle is again filled with methanol and sealed. The XAD-2® is allowed to soak indefinitely until the resin is need for sampling.
3. Decant the methanol and fines. Enough methanol is transferred to the bottle to cover the XAD-2® and the bottle is shaken vigorously.
4. The XAD-2® mixture is poured into the continuous extractor, just to the bottom of the 71/60 ground glass joint. The enlarging adapter with the glass frit is attached to the top of the extractor (it is important to wrap the outside of the 71/60 joint on the adapter with Teflon tape to achieve a good seal between the adapter and the extractor). A condenser is fitted to the top of the adapter to prevent solvent loss due to evaporation.
5. A 5 liter round bottom flask is filled with 4 L of methanol. The round bottom flask is heated and the methanol is distilled into the bottom of the continuous extractor. The distilled methanol flows up the extractor and back into the round bottom flask. The process continues for 18 hours. The methanol is removed from the round bottom flask and the extractor. The 5 L round bottom is filled with 4 L of methylene chloride and the extraction process continues.

It is recommended that two identical setups be employed, one for methanol and one for methylene chloride. If two are used, the methanol is removed from the extractor and the XAD-2® continuous extractor is attached to the methylene chloride apparatus and the extraction process continues for 18 hours. The methylene chloride in the round bottom flask is replaced whenever the distillation temperature shows evidence of contamination (typically, a rise in methylene chloride boiling point) or as needed, whenever several batches of XAD-2 have been cleaned without a full change in the source container of

methylene chloride.

The methylene chloride is drained from the extractor. A source of nitrogen is attached to a carbon filter in line to the inlet hose of the extractor. The nitrogen is turned on and the flow rate adjusted to approximately 1L per minute. The drying tower is checked periodically to assure that the clean XAD-2® does not fluidize in the bed. The resin is dried at this nitrogen flow rate for 12 hours. Heat tape wrapped around the extractor during the drying process will help prevent condensation in the XAD-2®.

The dried resin is transferred to appropriately sized glass bottles and labeled. The resin is then QC'd by MS for target analytes, tentatively identified compounds (TICs) and residual methylene chloride. An aliquot of the resin, approximately the same size as that used in a sampler, is extracted in the same manner as a sample blank. The extract is analyzed by the method that the XAD-2® sample extracts will be analyzed. The resin is ready to use if there are no interference peaks at or above its detection limit. The results of the analysis are documented in a notebook designated for XAD-2® preparation.

Note:

Toluene has been used as an additional extraction solvent in cases where new XAD-2 resin has been purchased from the manufacturer and the resin is suspected to be very "dirty" and contaminated. Typically the toluene extraction step is included before a methanol and two methylene chloride extraction steps. The toluene residue has proved to be very difficult to remove from the resin material after the extraction steps. Toluene is not necessary in cases where previously used XAD-2 is being recleaned for sampling.

SAMPLE PREPARATION

This LOI describes the procedure for sample extraction and preparation of samples collected on XAD-2® from BangBox tests.

1. A large Soxhlet apparatus, containing approximately 500 mL of methylene chloride and several boiling chips is assembled.
2. The PS-1 sample trap is placed in the soxhlet.
3. A method spike and a method blank are prepared in the same manner, using clean XAD-2® resin in place of the sampled resin. XAD-2® resin from the same batch as was used for the field samples is used.
5. 5 mL of the working lab surrogate solution (8270 surrogates) and 1 mL of the alternate working lab surrogate (d,-9,Phenylanthracene) solution is added to all of the samples, including field samples, blank and method spike.
6. The samples, blanks, and spikes are extracted for 18 hours.
7. The round bottomed flask is removed from the extractor.
8. Two boiling chips are placed in a 10 mL concentrator tube and the Kuderna-Danish (K-D) apparatus is assembled. A piece of pre-extracted glass wool is placed in a glass funnel and 20 g of anhydrous sodium sulfate is poured over the glass wool. The sample is dried by filtering through the sodium sulfate into the K-D inlet. The round bottomed flask is rinsed 3 times into the funnel with 10 mL aliquots of methylene chloride.
9. A 3-ball macro Snyder column is prewetted with methylene chloride and attached to the K-D inlet. The entire apparatus is placed on the water bath at 85°C. The concentrator tube is half immersed in the water bath, and the flask is bathed in water vapor. The apparatus is removed from the water bath when an apparent volume of 5 mL is observed in the concentrator tube. The solvent is allowed to drain for 5 minutes. The Snyder column is rinsed with 1 mL of methylene chloride and allowed to drain into the concentrator tube.

10. The concentrator tube is removed from the K-D evaporator. A 2-ball macro Snyder column is attached to the concentrator tube and prewetted with 2 mL of methylene chloride. The apparatus is placed on the water bath and the sample concentrated to 2 mL. The Snyder column is removed and the volume is brought up to 5 mL with methylene chloride. If required, a volume other than 5 mLs may be used as a final sample volume for specifically designated samples.
11. The sample is transferred to a 7.5 mL vial with a teflon lined screw top and stored in a refrigerator at 4°C. An analysis aliquot of 1 mL is removed from the 5 mL vial and transferred to a 2 mL crimp-top glass vial and labeled.

GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

This LOI describes the GC/MS instrumentation and methodology for analysis of extracts of OB/OD samples. Full scan electron ionization mass spectrometry (EI/MS) will be used to identify and quantitate targeted analytes and to identify and semi-quantitate untargeted analytes.

A. EI/MS

1. The instrument used for this method is a Hewlett Packard GC/MS operated in a conventional GC/MS mode.
2. The analytical method is based on U.S. EPA SW-846 Method 8270, Test Methods for Evaluating Solid Waste, 3rd Edition, September 1986. Method 8270 must be read and understood by the GC/MS analyst before beginning any semi-volatiles analysis.
3. Lists of instrument operating conditions for the Hewlett Packard in use for Method 8270 are shown in Tables 1 and 2.
4. The instrument is tuned and mass calibrated as required using perfluorobutylamine (FC-43). Mass calibration is performed using procedures provided by the instrument manufacturer. The tune is verified by injecting 50 ng of decafluorotriphenylphosphine (DFTPP) and checking the ion abundance criteria against those listed in Table 3. If the DFTPP mass spectrum does not meet specifications, the DFTPP is reanalyzed or the mass spectrometer is re-tuned so that it will meet the criteria. The DFTPP tune must be met before calibration or sample analysis can begin. The DFTPP tune is valid only for 12 hours; DFTPP is analyzed every 12 hours.

B. CALIBRATION PROCEDURES

1. Method 8270 calibration procedures and criteria apply with the exceptions of the lengthened GC program (Table 2) and the shortened target list (Table 4). Designated calibration check compounds and system performance check compounds must meet the criteria outlined in Method 8270.
2. Existing data system procedures automate the quantitation procedures and generate a system response curve.

3. Calibration is verified daily (or every 12 hours) by analyzing a mid-level calibration solution in accordance with Method 8270 specifications.

C. SAMPLE ANALYSIS

1. A solvent blank is analyzed prior to sample analysis to demonstrate that the analytical system is free from contamination.
2. The internal standard area counts for each sample analysis are verified to be between 50% and 150% of the last daily calibration standard, in accordance with Method 8270 specifications.
3. Surrogate recoveries for each sample are checked against Method 8270 surrogate spike recovery limits for soil/sediment samples. Surrogate recoveries from the XAD-2® and the sample matrix used in this work have not been determined experimentally. Samples will be re-analyzed if surrogate recoveries fall below the levels specified in Method 8270.
4. In order to gauge recoveries of target compounds from XAD-2®, two samples will be generated in the laboratory by spiking clean XAD-2® with selected target compounds and surrogates (Table 4). These samples will be subjected to a similar workup to the samples generated in the field.
5. The criteria for identification of target spectra are positive matching of the relative retention times and the spectra of the sample and the standard components in accordance with Method 8270 specifications.
6. Quantitation is achieved by the use of automated procedures in the Hewlett Packard Data System.
7. Several nonhalogenated compounds (listed below) found on the 8270 list do not chromatograph well. Although these compounds are calibrated and reported, quantitative results in the samples are flagged. These compounds include:

1,2-Diphenylhydrazine
 α,α -Dimethylphenethylamine
p-Dimethylaminoazobenzene
4-Aminobiphenyl
Ethyl methanesulfonate
Dibenz(a,j)acridine
Benzidine

D. ANALYSIS SCHEME

1. Samples extracts which are scheduled to be exchanged with other laboratories for QC purposes will be spiked with 1-nitronaphthalene prior to analysis.
2. If the target compounds are detected at low levels (20-60 ng/uL) or if the target compounds are not detected at all, samples may be concentrated to an accurate volume between 1 mL and 2.5 mL for analysis.

E. QUALITATIVE/SEMI-QUANTITATIVE ANALYSIS

1. Mass spectral interpretation of non-target compounds is conducted using automated data reduction programs. Compound identifications are verified manually by experienced interpreters of mass spectral data and compared to the reference library (NBS) spectrum in the most concentrated samples.
2. Automated semi-quantitation is achieved by comparison of the peak area of the non-targeted compound to that of the closest eluting standard.

F. LABORATORY NOTEBOOKS

1. A designated laboratory notebook is used. All notebook entries are made in accordance with the Radian Corporation Standard Operating Procedure For Maintaining Laboratory Notebooks, No. 313-AD-012.
2. All samples are recorded in a sample logbook.

G. DATA REPORTING AND ARCHIVAL

1. All sample data are reduced, reviewed and reported according to the Radian Corporation Standard Operating Procedure for Data Reduction and Data Review in the GC/MS laboratory, No. 313-MS-041. The data package includes calibration curve, DFTPP tune, daily calibration, blank and sample data. A total ion chromatogram and quantitation reports are included with each sample or calibration standard report. Enhanced spectra along with the five best library search results are included with the analytical reports for selected, most concentrated samples.
2. All sample data are archived and verified. All archive information is documented in the project notebook and data archive index for the

appropriate instrument.

3. Data reports will clearly show which samples are combined for composite GC/MS assay. At a minimum each data table will show Radian ID number, munition, sample volumes for the PS-1 samplers, the Method 8270 analytes, the estimated PQL based on Method 8270, and the mass of analyte per total volume of air sampled by the PS-1 samplers.

TABLE 1. MASS SPECTROMETER PARAMETERS

Mass Spectrometer:	Hewlett Packard
Data System:	EnviroQuant
Manifold Temperature:	100°C
Scan Mode:	Full Scan
Mode:	Electron ionization
Electron Energy:	70 eV
Resolution:	Unit
Interface Temperature:	270°C
Mass Range:	35-500 amu
Scan Rate:	1 scan/sec

TABLE 2. GC PARAMETERS

Column:	60 m x 0.32 mm I.D., DB-5 or DB-1 fused silica capillary column, or equivalent, 1.0 um film
Carrier Gas:	Helium 1 mL/min
Injector:	Split/splitless
Injector Temperature:	280°C
Interface:	Direct source coupled
Injection Volume:	1-2 uL
GC Program:	Initial hold of 5 min. @ 5°C - 300°C @ 10°/min, hold 30 min.

TABLE 3. DFTPP KEY IONS AND ION ABUNDANCE CRITERIA^a

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

^aJ.W. Eichelberger, L.E. Harris, and W.L. Budde. "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography-Mass Spectrometry", *Analytical Chemistry*, **47**, 995 (1975).

TABLE 4. Target Compound List

Compound	
acenaphthene (CCC)	methyl methanesulfonate
acenaphthylene	2-methylnaphthalene
acetophenone	2-methylphenol
anthracene	4-methylphenol
benzo(a)anthracene	naphthalene
benzo(b)fluoranthene	1-naphthylamine
benzo(k)fluoranthene	2-naphthylamine
benzo(g,h,i)perylene	2-nitroaniline
benzo(a)pyrene (CCC)	3-nitroaniline
benzyl alcohol	4-nitroaniline
butyl benzyl phthalate	2-nitrophenol (CCC)
chrysene	4-nitrophenol (SPCC)
dibenz(a,h)anthracene	perylene
dibenzofuran	phenacetin
di-n-butyl phthalate	phenanthrene
diethyl phthalate	phenol (CCC)
7,12-dimethylbenz(a)anthracene	phenylanthracene-d ₉ (*)
2,4-dimethylphenol	1-nitronaphthalene (*)
dimethyl phthalate	1,4-dichlorobenzene-d ₄ (IS)
4,6-dinitro-2-methylphenol	naphthalene-d ₈ (IS)
2,4-dinitrophenol (SPCC)	acenaphthene-d ₁₀ (IS)
2,4-dinitrotoluene	phenanthrene-d ₁₀ (IS)
2,6-dinitrotoluene	chrysene-d ₁₂ (IS)
diphenylamine	perylene-d ₁₂ (IS)
di-n-octyl phthalate (CCC)	2-fluorophenol (SURR)
fluoranthene (CCC)	phenol-d ₆ (SURR)
fluorene	nitrobenzene-d ₅ (SURR)
indeno(1,2,3-cd)pyrene	2-fluorobiphenyl (SURR)
isophorone	2,4,6-tribromophenol (SURR)
2-methylcholanthrene	terphenyl-d ₁₄ (SURR)

KEY: IS = Internal Standard
 SURR = Surrogate
 * = Additional Internal Standards for Extract Exchanges
 CCC = Calibration Check Compounds
 SPCC = System Performance Check Compounds

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SAMPLE CUSTODY AND IDENTIFICATION

1) SAMPLE IDENTIFICATION, TRACKING, AND STORAGE

Since the results from the analytical methods are to be evaluated statistically to determine the appropriateness of the application of the test methodology, standard procedures must be implemented to ensure that the samples are correctly identified and their integrity not compromised prior to analysis. Laboratory standard operating procedures for sample preparation, handling, tracking, and storage must be followed.

To ensure that sample identification, tracking, and storage procedures are adequate to ensure sample integrity, the following procedures will be followed during the sampling and analysis efforts related to the BangBox testing:

- Unique identification numbers will be assigned to each sample generated and the number used or referenced throughout sample analysis; Sample number will incorporate Julian dates to track sampling dates consistently throughout the project;
- Sample containers and tops will be selected based on their non-reactivity with the sample matrix;
- Sample labels will include sufficient information to avoid sample mix-ups and assess sample hold times;
- The temperature of the sample storage environment will be monitored to ensure that it is maintained within an acceptable storage temperature range; and
- The retrieval and return of the sample to the storage area will be recorded in the storage area log books.
- In cases where samples are shipped by overnight carrier or transported in any way, proper care will be taken to preserve the temperature of the sample storage environment. The samples will be carefully labeled and packed to survive shipping conditions.

- Specific labeling and sample numbering activities are described in greater detail in the analysis-specific letters of instruction and in the QA/QC letters of instruction (LOI 10).

Project data and procedural audits will help to ensure that these procedures are followed or modified as needed to ensure the integrity of the samples during the conduct of the work.

DIOXIN AND FURAN SAMPLING AND ANALYSIS

1) SAMPLE COLLECTION, ANALYSIS, DATA REPORTING

Dioxin/furan sampling will be conducted using a single, dedicated PS-1 sampler and following the LOI 26 instructions. The following differences between traditional PS-1 sampling with XAD-2 and the dioxin/furan sampling are important to note:

1) One sampler will be used for the dioxin/furan sampling. This sampler is used in addition to the other PS-1 samplers designated for SVOC sampling.

2) The dioxin/furan sampling is to be conducted only when chlorinated munition items are scheduled for detonation in the BangBox. These munitions will be designated in advance and are currently planned for approximately half of the 10-Item test materials.

3) Because of the specialized nature of the XAD-2 sampling for dioxins/furans, the XAD-2 will be prepared especially for the dioxin sampling. The resin cartridges will be spiked with internal standard compounds by the analytical laboratory as specified in LOI 36. For the purposes of the 10-Item Testing in Dugway, the prespiked XAD-2 cartridges will be cleaned and prepared in North Carolina and shipped to the site for use in dioxin/furan sampling. Sampled cartridges will then be shipped back to North Carolina for analysis. The cartridges will be solvent extracted and the extracts will be solvent exchanged, taken through column cleanup procedures, spiked with recovery standards and analyzed for PCDDs and PCDFs by High Resolution GC/MS in Selected Ion Monitoring (SIM) mode.

4) The dioxin/furan XAD-2 cartridges will be handled separately and shipped for analysis in separately labeled containers with unique sample numbers to ensure that the dioxin/furan spiked cartridges are handled, sampled, and analyzed properly.

It is important to note that PS-1 cartridges for dioxin analysis will be shipped to the field pre-spiked and filled (loaded) with XAD-2 that is designated for dioxin sampling. However, PS-1 cartridges for SVOC analysis will be shipped to the field empty and filled from a clean container of XAD-2 as needed for sampling in the PS-1 SVOC samplers.

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DETERMINATION OF HYDROGEN CHLORIDE SAMPLING PROCEDURE

1) APPLICABILITY, PRINCIPLE, INTERFERENCES, PRECISION, BIAS, AND STABILITY

APPLICABILITY. This method is applicable for determining emissions of hydrogen chloride (HCl) and chlorine (Cl₂) from stationary sources.

PRINCIPLE. An integrated sample is extracted from the source and passed through a prepurged heated filter into dilute sulfuric acid and dilute sodium hydroxide solutions which collect the gaseous hydrogen chloride and chlorine, respectively. The filter collects other particulate matter including halide salts. The hydrogen chloride is solubilized in the acidic solution to form chloride (Cl⁻) ions. The chlorine has a very low solubility in the acidic solution and will pass through to the alkaline solution where it is hydrolyzed to form a proton (H⁺), the halide ion, and the hypohalous acid (HClO). Sodium thiosulfate is added in excess to the alkaline solution to assure reaction with the hypohalous acid to form a second halide ion such that 2 halide ions are formed for each molecule of halogen gas. The halide ions in the separate solutions are measured by ion chromatography (IC).

INTERFERENCES. Volatile materials, such as chlorine dioxide (ClO₂) and ammonium chloride (NH₄Cl), which produce halide ions upon dissolution during sampling are potential interferents. Interferents for the halide measurements are the halogen gases which disproportionate to a hydrogen halide and a hydrohalous acid upon dissolution in water. However, the use of acidic rather than neutral or basic solutions for collection of the hydrogen halides greatly reduces the dissolution of any halogens passing through this solution. The simultaneous presence of HBr and Cl₂ may cause a positive bias in the HCl result with a corresponding negative bias in the Cl₂ result as well as affecting the HBr/Br₂ split. High concentrations of nitrogen oxides (NO_x) may produce sufficient nitrate (NO₃⁻) to interfere with measurements of very low Br⁻ levels.

PRECISION AND BIAS. The within-laboratory relative standard deviations are 6.2 and 3.2 percent at HCl concentrations of 3.9 and 15.3 ppm, respectively. The method does not exhibit a bias to Cl_2 when sampling at concentrations less than 50 ppm.

SAMPLE STABILITY. The collected Cl_2 samples can be stored for up to 4 weeks.

DETECTION LIMIT. The analytical detection limit for Cl_2 is $0.1 \mu\text{g/ml}$. Detection limits for the other analyses should be similar.

2) APPARATUS

SAMPLING. The component parts of the sampling train are discussed briefly below.

FILTER. A 47-mm Teflon-glass mat, Pallflex TX40HI75 (Pallflex Inc., 125 Kennedy Drive, Putnam, CT 06260). This filter is in a mat configuration to prevent fine particulate matter from entering the sampling train. Its composition is 75% Teflon/25% borosilicate glass.

Other filters may be used, but they must be in a mat (as opposed to a laminate) configuration and contain at least 75% Teflon. Filters will be archived for potential halide salts analyses.

IMPINGERS. Six 30-ml midget impingers with leak-free glass connectors.

FILTER HOLDER AND SUPPORT. The filter holder should be made of Teflon. The filter support shall be made of Teflon.

SAMPLE LINE. Leak-free, with compatible fittings to connect the last impinger to the needle valve.

RATE METER. Rotameter, or equivalent, capable of measuring flow rate to within 2 percent of the selected flow rate of 2 liters/min.

TEMPERATURE MEASURING DEVICES. Temperature measuring device to monitor the temperature of the filter and a thermometer or other temperature measuring device to monitor the temperature of the sampling system from the outlet of the filter to the

inlet of the first impinger.

ICE WATER BATH. To minimize loss of absorbing solution.

3) REAGENTS

Unless otherwise indicated, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society (ACS reagent grade). When such specifications are not available, the best available grade shall be used.

SAMPLING.

Water. Deionized, distilled water that conforms to ASTM Specification D 1193-77, Type 3.

Acidic Absorbing Solution, 0.1 N Sulfuric Acid (H_2SO_4). To prepare 100 ml of the absorbing solution for the front impinger pair, slowly add 0.28 ml of concentrated H_2SO_4 to about 90 ml of water while stirring, and adjust the final volume to 100 ml using additional water. Shake well to mix the solution.

Alkaline Adsorbing Solution, 0.1 N Sodium Hydroxide (NaOH). To prepare 100 ml of the scrubber solution for the fourth impinger, dissolve 0.40 g of solid NaOH in about 90 ml of water, and adjust the final solution volume to 100 ml using additional water. Shake well to mix the solution.

Sodium Thiosulfate ($Na_2S_2O_3 \cdot 5 H_2O$)

SAMPLE PREPARATION AND ANALYSIS.

Water. Deionized, distilled water that conforms to ASTM Specification D 1193-77, Type 3.

Absorbing Solution Blanks. A separate blank solution of each absorbing reagent should be prepared for analysis with the field samples. Dilute 30 ml of each absorbing solution

to approximately the same final volume as the field samples using the blank sample of rinse water.

Halide Salt Stock Standard Solutions. Prepare concentrated stock solutions from reagent grade sodium chloride (NaCl). This must be dried at 110°C for two or more hours and then cooled to room temperature in a desiccator immediately before weighing. Accurately weigh 1.6 to 1.7 g of the dried NaCl to within 0.1 mg, dissolve in water, and dilute to 1 liter. Calculate the exact Cl⁻ concentration using Equation 1.

$$\mu\text{g Cl}^-/\text{ml} = \text{g of NaCl} \times 10^3 \times 35.453/58.44 \quad \text{Eq. 1}$$

Alternately, solutions containing a nominal certified concentration of 1000 mg/l NaCl are commercially available as convenient stock solutions from which standards can be made by appropriate volumetric dilution. Refrigerate the stock standard solutions and store no longer than one month.

4) PROCEDURE

SAMPLING.

Preparation of Collection Train. Prepare the sampling train as follows: Pour 15 ml of the acidic absorbing solution into each one of the first pair of impingers, and 15 ml of the alkaline absorbing solution into each one of the second pair of impingers. Connect the impingers in series with the knockout impinger first, if used, followed by the two impingers containing the acidic absorbing solution and the two impingers containing the alkaline absorbing solution. Place a fresh charge of silica gel in the impinger at the end of the impinger train.

Adjust the temperature of the filter to 150° F in order to prevent water condensation in the filter holder. The temperature should be monitored throughout a sampling run to ensure that the desired temperature is maintained.

Leak-Check Procedure. A leak-check prior to the sampling run is optional; however, a leak-check after the sampling run is mandatory. The leak-check procedure is as follows: Temporarily attach a suitable (e.g., 0-40 cc/min) rotameter to the outlet of the dry gas meter and place a vacuum gauge at or near the probe inlet. Plug the filter

inlet, pull a vacuum of at least 250 mm Hg (10 in. Hg), and note the flow rate as indicated by the rotameter. A leakage rate not in excess of 2 percent of the average sampling rate is acceptable. (NOTE: Carefully release the probe inlet plug before turning off the pump.) It is suggested (not mandatory) that the pump be leak-checked separately, either prior to or after the sampling run. If done prior to the sampling run, the pump leak-check shall precede the leak-check of the sampling train described immediately above; if done after the sampling run, the pump leak-check shall follow the train leak-check. To leak-check the pump, proceed as follows: Disconnect the drying tube from the probe-impinger assembly. Place a vacuum gauge at the inlet to either the drying tube or pump, pull a vacuum of 250 mm (10 in.) Hg, plug or pinch off the outlet of the flow meter, and then turn off the pump. The vacuum should remain stable for at least 30 sec.

Sample Collection. Turn on the sampling pump to permit gas to be pulled through the impinger train. Adjust the sampling rate to 2 liters/minute, as indicated by the rate meter, and maintain this rate to within 10 percent during the entire sampling run. A calibrated orifice will be used to maintain a constant sampling rate of 2 liters/minutes. A sampling time of one hour is recommended. Shorter sampling times may introduce a significant negative bias in the HCl concentration. At the conclusion of the sampling run, perform a leak-check as described above.

SAMPLE RECOVERY. Disconnect the impingers after sampling. Quantitatively transfer the contents of the acid impingers and the knockout impinger, if used, to a leak-free storage bottle. Add the water rinses of each of these impingers and connecting glassware to the storage bottle. Repeat this procedure for the alkaline impingers and connecting glassware using a separate storage bottle. Add 25 mg sodium thiosulfate per the product of ppm of halogen anticipated to be in the stack gas times the dscm stack gas sampled. [Note: This amount of sodium thiosulfate includes a safety factor of approximately 5 to assure complete reaction with the hypohalous acid to form a second Cl⁻ ion in the alkaline solution.] Save portions of the absorbing reagents (0.1 N H₂SO₄ and 0.1 N NaOH) equivalent to the amount used in the sampling train (these are the absorbing solution blanks described in Section 3.2.2); dilute to the approximate volume of the corresponding samples using rinse water directly from the wash bottle being used. Add the same amount of sodium thiosulfate solution to the 0.1 N NaOH absorbing solution blank. Also, save a portion of the rinse water used to rinse the sampling train.

Place each in a separate, prelabeled storage bottle. The sample storage bottles should be sealed, shaken to mix, and labeled. Mark the fluid level.

5) CALIBRATION

DRY GAS METERING SYSTEM, THERMOMETERS, RATE METER, AND BAROMETER. Same as in EPA 40 CFR Subpart 60, Appendix A, Method 6, Sections 5.1, 5.2, 5.3, and 5.4.

6) CALCULATIONS

Retain at least one extra decimal figure beyond those contained in the available data in intermediate calculations, and round off only the final answer appropriately.

SAMPLE VOLUME, DRY BASIS, CORRECTED TO STANDARD CONDITIONS.
Calculate the sample volume using Eq. 6-1 of Method 6.

TOTAL μg HCl PER SAMPLE.

$$m_{\text{HX}} = K V_s (S_{\text{X}^-} - B_{\text{X}^-}) \quad \text{Eq. 4}$$

where: B_{X^-} = Mass concentration of applicable absorbing solution blank, μg halide ion (Cl^-)/ml, not to exceed 1 $\mu\text{g}/\text{ml}$ which is 10 times the published analytical detection limit of 0.1 $\mu\text{g}/\text{ml}$.

m_{HX} = Mass of HCl in sample, μg .

S_{X^-} = Analysis of sample, μg halide ion (Cl^-)/ml.

V_s = Volume of filtered and diluted sample, ml.

K_{HCl} = 1.028 (μg HCl/ μg -mole)/(μg Cl^- / μg -mole).

TOTAL μg Cl_2 or Br_2 PER SAMPLE.

$$m_{\text{X}_2} = V_s (S_{\text{X}^-} - B_{\text{X}^-}) \quad \text{Eq. 5}$$

where: m_{X_2} = Mass of Cl_2 in sample, μg .

CONCENTRATION OF HYDROGEN HALIDE OR HALOGEN IN FLUE GAS.

$$C = K m_{HX, X_2} / V_{m(std)} \quad \text{Eq. 6}$$

where: C = Concentration of hydrogen halide (HX) or halogen (X_2), dry basis, mg/dscm.

$V_{m(std)}$ = Dry gas volume measured by the dry gas meter, corrected to standard conditions, dscm.

K = 10^{-3} mg/ μg .

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1. DATA REDUCTION AND ANALYSIS - OUTLINE OF PROCEDURE

2. DATA RECEIVED

2.1. Results from: (1) individual sample assays (wt/vol, wt/area, etc.), (2) all blanks (laboratory method blanks, travel blanks, field blanks, etc.), (3) control standards (field standards, storage controls, sample preparation controls, laboratory quality control standards, etc.), and (4) duplicate assays. All these results are to be presented in the chronological order in which the samples were analyzed, along with notations as to when instrument conditions were changed, equipment maintenance performed, etc.

2.2. Daily calibration curves and all data used to generate the line of best fit. If the mean of several separate assays were used as the input data, furnish individual assay results. Describe the standard solutions, e.g., whether they include only a single compound or several compounds.

2.3. Algorithms (with constants) that are used in computing results in terms of engineering units from the measured units; e.g., wt/vol at standard conditions from millivolts.

2.4. Real-time data sampled and recorded in 5-s intervals for periods of time and instruments specified in the detailed test plan. Some instruments (e.g. thermocouples) will require sampling and recording of data more frequently, e.g., every 0.2 s. Data will be recorded in an ASCII file that can be imported into a LOTUS 1-2-3 file with column and row identification, and with units. All calibration and daily span check data will also be recorded in the same format.

2.5. Weights of cleaned quartz and Teflon[®] filters, and of the same filters after sampling will be furnished for each sample prior to extraction and assay. All weights will be to an accuracy that is traceable to a certified weight standard and to a precision as specified in the detailed test plan.

2.6. All data produced from chemical analyses and other measurements. (The operator is not to arbitrarily delete readings.) Any data collector or other worker on the project is to identify any unusual incident and record special observations pertinent to sample collection, handling, storage, and analysis, and/or to the conduct of the test, associating the remarks with an individual sample whenever possible. Submit this information immediately upon completion of a task.

2.7. All log books from the test or a copy of all the pages that refer to the chlorine test will be furnished as part of the data.

3. DATA ANALYSIS

3.1. Examine graphic output of real-time instruments after each trial, to determine if the trial was successfully sampled and recorded and if corrective action is required before the next trial.

3.2. Verify the unit conversion algorithms by converting at least one non-zero value (in measured units) to engineering units.

3.3. Correct all analyte concentrations for measured background levels, as required.

3.4. Calculate the emission factor for each measured emission. Using these emission factors from each sampling point, determine the total mass of each element and chemical compound from the detonation or burn.

3.5. Compare the concentrations of CO and CO₂ derived from the 6-L canister sampling system with the concentrations derived from the real-time sampling system.

3.6. Calculate (as data permits) all components of variance, e.g., between filter variation within trial (weight and chemical analysis), between subsamples of the same extract when analyzed as separate samples on different days etc. The amount of data available for these calculations will depend upon assets that the BangBox Test Director (TD) can obligate to the program.

3.7. All results from these analyses/comparisons will be furnished the TD and only upon his approval will additional distribution be made.

4. OUTLIER DATA

All available data and recorded operator comments will be considered prior to deleting a suspected outlier data point. If the comments do not provide a reasonable rationale for removing the datum, the laboratory worker or other data collector will be asked to review the information and make further remarks. All data points deleted by the statistical analyst will be documented, together with the reason for the deletion.

APPENDIX L. LETTER OF INSTRUCTION

ECO, L.C.

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APPENDIX M. DISTRIBUTION LIST

TO BE PROVIDED

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