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HEADQUARTERS
U.S. ARMY ARMAMENT,
MUNITIONS AND CHEMICAL COMMAND

FINAL REPORT

DEVELOPMENT OF METHODOLOGY AND TECHNOLOGY FOR IDENTIFYING AND QUANTIFYING EMISSION PRODUCTS FROM OPEN BURNING AND OPEN DETONATION THERMAL TREATMENT METHODS.

BANGBOX TEST SERIES

VOLUME 1
TEST SUMMARY

JANUARY 1992

Maintenance Management Division
Demilitarization and Technology Branch
Rock Island, Illinois 61299-6000
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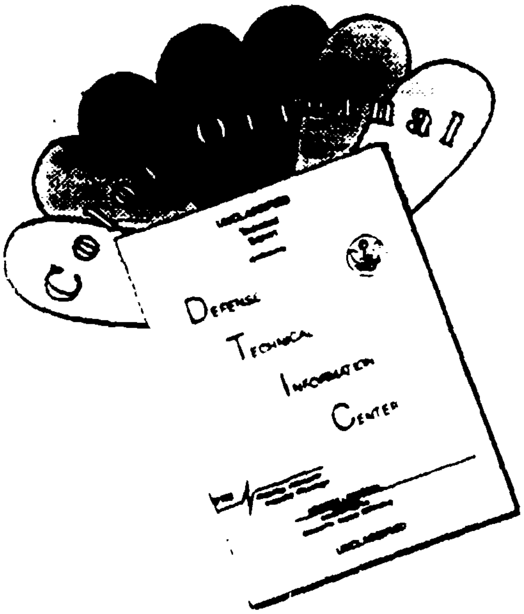

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The report addresses the gaseous and particulate sampling of TNT detonations and double-base and manufactures residue propellant burns. All trials were accomplished in an air supported building (BangBox). Sampling was accomplished with real time instruments for CO_2, CO, NO_x, O_3, and SO_2; canister samplers for SF_6, CO_2, CO, and volatile hydrocarbons; Quartz fiber filters with backup XAD-2™ resin cartridges for semivolatile organics, and various instruments for size and mass of particles. For each analyte measured an emission factor was calculated by the concentration times volume of the building and by the carbon balance method. The carbon balance method is based on the conservation of carbon in the detonation or burn and thus becomes the trace element used as the basis for determining emission factors. The mass balance of carbon in the TNT detonation was established in the BangBox with CO_2 contributing 97 percent of the total carbon, CO carbon (0.50 percent), semivolatile and non volatile organics carbon (0.57 percent), and soot (1.7 percent).
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The tracer method (carbon balance) permits an emission factor estimate from every parcel of sampled air regardless of the homogeneity of the cloud whereas the concentrations times cloud volume method is only good when the concentration is constant throughout the entire cloud volume and the cloud volume can be measured. The test was successful in providing a method for obtaining emission factors in field testing, providing data to select samplers, and the effectiveness of the supercritical fluid chromatography assay for field detonations and burns of PEP material.

FOREWORD

A test planning directive to conduct the OB/OD test in support of U.S. Army Armament, Munitions and Chemical Command (AMCCOM) was issued by U.S. Army Test and Evaluation Command (TECOM) on 28 April 1988[1]. This test was conducted following the Technical Steering Committee Symposium which was convened in July 1988. The requirement for identifying and quantifying emissions from the open detonation of explosives and open burning of propellants was discussed in detail by authorities from throughout the military, academic, and commercial communities. Conclusions and recommendations developed during the symposium are reported in proceedings of the symposium[2].

The BangBox Test series report includes three volumes:

Volume 1. A summary which describes the planning phase, the conduct of trials, sample analyses and results, and the conclusions and recommendations. It is useful for those who need the background, synopsized results, conclusions, and recommendations without the complete details with the supporting data and information.

Volume 2. A stand-alone document which covers the detail of the complete test. It describes the test development, description of the test materiel, and the trial results as they relate to the test objectives and the explosives and propellants tested.

Volume 3. The quality assurance/quality control (QA/QC) report covers the QA/QC plan, detailed test plan, the letters of instruction (prepared for procedural instruction), the quality audits, the reports of the quality audits, and the results of the blind spikes analyzed by the laboratories.

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[1] Letter, AMSTE-TA-F, Headquarters, U.S. Army Test and Evaluation Command, Aberdeen Proving Ground, Maryland, 20 April 1988, subject: Test Planning Directive for Special Study of Open Burning/Open Detonation (OB/OD), Phase II, TECOM Project No 2-CO-210-000-017.

[2] *Proceedings of the Technical Steering Committee Symposium 6-8 July 1988*, Headquarters, United States Army Armament, Munitions and Chemical Command, Rock Island, Illinois, August 1991.

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Mr. Dean Sevey, U.S. Army Armament, Munitions and Chemical Command served as a member of the U.S. Army Materiel Command (AMC) Management Steering Committee.

The U.S. Environmental Protection Agency (EPA) provided technical guidance and support during both the test planning and execution phases of this test, review of data collection and analytical procedures, and assurance of instrument accuracy. Mr. Chester Oszman, Office of Solid Waste, Washington, D.C., provided programmatic and technical guidance to the AMCCOM Program Manager. Dr. William Mitchell, Mr. Raymond Rhodes, and the staff of the Atmospheric Research and Exposure Assessment Laboratory, Research and Monitoring Evaluation Branch, Research Triangle Park, North Carolina, provided planning guidance, quality assurance/quality control support, and field and laboratory monitoring and audit support.

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Andrulis Research Corporation was responsible for symposium conduct, test plan preparation, overall data analysis, and preparation of this final report. Data analysis and plan and report writing were accomplished by Mr. Cecil Eckard, Dr. Kenneth Zahn, Mr. Douglass Bacon, Mr. Duane Long, Mr. A. Lacy Hancock, and Mr. Joseph Kohlbeck. Ms. Dorothy Arnold and Ms. Linda Chastain provided technical editing, and Ms. Teresa Jensen and Ms. Cheri Martens provided administrative support.

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Mr. Robert A. Cary of Sunset Laboratory, Forest Grove, Oregon, provided thermal analysis of filter samples for organic, elemental, and inorganic carbon.

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ABSTRACT

Open burning (OB) and open detonation (OD) are currently the primary means of demilitarization employed by the Department of Defense (DoD) for the treatment of explosive residue, propellants, and munitions as they become unsafe, excess, obsolete, or unrepairable. Increasingly stringent requirements for environmental documentation of potential pollution/contamination from combustion products under such acts as the Clean Air Act, Clean Water Act, and Resource Recovery Act have resulted in a critical need for a test program to collect data to be used as a basis for informed decisions concerning the limitations/restrictions of OB/OD, the need for alternative methods where required, and maintaining an effective, economical, and environmentally safe means of accomplishing the required demilitarization/treatment.

Under the sponsorship of the Single Manager for Conventional Ammunition within the DoD, a symposium was conducted in July 1988[3] to develop planning concepts necessary to address the technical problems associated with an accurate environmental characterization of the OB/OD processes. Authorities from governmental, academic and private research organizations discussed the technical issues and concepts of testing, and the associated sampling and sample analysis technologies, data analysis processes, test organization, and preparation of reports that would be accepted by Federal and State regulatory agencies. Expertise represented included field sampling, instrumentation, field and laboratory analysis, environmental documentation, atmospheric dispersion, data processing, combustion and explosive phenomenology, and quality assurance/quality control. A technical steering committee (TSC) composed of recognized experts in their respective disciplines was formed under the leadership of the U.S. Army Armament, Munitions and Chemical Command Program Manager.

A list of volatile and semivolatile organic compounds and metals which are potential contaminants in either the soil or atmosphere from OB/OD processes was developed. A chamber (BangBox (BB)) test was conducted at Sandia National Laboratories to check out instrumentation, technology, methodology, and analytical procedures that were proposed for follow-on field tests to

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[3] *Proceedings of the Technical Steering Committee Symposium 6-8 July 1988,* Headquarters, United States Army Armament, Munitions and Chemical Command, Rock Island, Illinois, August 1991.

be sampled by the tested instrumentation placed on a fixed-wing aircraft flying through the plume. The field tests are required to obtain data to validate the technology and methodology for characterizing full scale OB/OD operations and establish correlations between the BB and full scale operations. If correlations can be established the less expensive BB type of testing may be used for emission characterization of various munitions and explosives in the demilitarization inventory. The U.S. Environmental Protection Agency (EPA) provided technical guidance and support during test planning and execution phases of test, review of data collection and analytical procedures, and assurance of instrument accuracy. Real time and near real time particulate and gaseous concentration measurements were achieved. These data were correlated with the samples collected on filters and gaseous containers and held for subsequent laboratory analysis. A methodology of using carbon balance to calculate more accurate emission factors of combustion products in diffusing clouds was verified.

The BB tests evaluated emission factors from 2,4,6-trinitrotoluene, OD and double- and composite-based propellants, OB. The tests confirmed the technologies, methodologies, and analytical procedures employed. These processes will be the basis for collecting and analyzing the data from the followon large-scale open-air tests scheduled for Phases A, B, and C to be conducted between June 1989 and September 1990. Further refinements will be made as required and approved by the TSC.

SECTION 1. INTRODUCTION

1.1. Open burning (OB) and open detonation (OD) are currently the primary means of demilitarization employed by the Department of Defense (DoD) for the treatment of explosive residue, propellants, and munitions as they become unsafe, excess, obsolete, or unrepairable.

1.2. The increasing need for data on OB/OD combustion products to support environmental documentation requirements, such as those of the Clean Air Act (CAA), Clean Water Act (CWA), and Resource Conservation and Recovery Act (RCRA) resulted in a critical need for a test program to collect data to be used as a basis for informed decisions concerning the limitations/restrictions of OB/OD, the need for alternative methods where required, and maintaining an effective, economical, and environmentally safe means of accomplishing the required demilitarization/treatment. The Single Manager for Conventional Ammunition within the DoD began to address this need in the early 1980's, and to provide scientific data to answer the question of environmental acceptability of OB/OD thermal treatment methods.

1.3. Although limited data are available from past studies on the generation of particulates and criteria pollutants from small-scale laboratory and field OB/OD operations, little field data are available on the levels of semivolatile organic emissions that result from unconstrained combustion of propellants by open-air burning or of explosives by open-air detonation. These compounds are difficult to collect and identify when produced in low concentrations. Thus, prior to the conduct of full-scale OB/OD field testing operations involving large quantities (thousands of kilograms) of explosives and propellants, a limited number of small-scale explosive detonation and burning trials were conducted within a controlled, ambient air environment. The test chamber used to provide this controlled environment was that operated by Sandia National Laboratories (SNL), Kirtland Air Force Base (AFB), New Mexico, and is locally known as the BB.

1.4. The purpose of this report is to describe the BB test technical issues, objectives, methodology development, conduct, data collection, analysis procedures, quality assurance/quality control procedures, results, and conclusions. At various places within the report or its appendices, this test is referred to as the BB, preliminary, or chamber test; these terms are considered equivalent.

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SECTION 2. TECHNICAL ISSUES

2.1. The Symposium

2.1.1. Background

OB/OD operations are conducted at a large number of DoD installations and activities. Each location has unique conditions of soil type, groundwater depth, vegetative cover, terrain, sensitivity to noise or airborne particulate levels, proximity to urban areas, and types of materials requiring demilitarization. Because of the common need of these installations and activities for high-quality data on combustion products to support permit applications and environmental documentation, the U.S. Army Armament, Munitions and Chemical Command (AMCCOM) OB/OD program manager established a technical steering committee (TSC) to provide advice and guidance on test planning, test conduct, and data analysis. In July 1988, a symposium was conducted in Salt Lake City, Utah, to develop the planning concepts necessary to address the many technical problems associated with conduct of a successful OB/OD test program. Authorities from governmental, academic, and private organizations discussed data requirements, sampling and sample analysis technologies, data analysis processes, test organization, and preparation of reports that would be accepted by Federal and State regulatory agencies. Disciplines represented at this symposium included test planning; field sampling; instrumental, field, and laboratory analysis; environmental documentation; atmospheric dispersion; data treatment; combustion and explosive phenomenology; and quality assurance/quality control (QA/QC). It was at this time that the TSC, composed of recognized experts in their respective disciplines, was formed. The results of this symposium are outlined in a separate report[4] and briefly summarized below.

2.1.2. Results.

2.1.2.1. The symposium recognized that open-air, surface detonations of explosives produce a short-term, high-temperature buoyant fireball of oxidizing gases which may entrain soil and which

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[4] *Proceedings of the Technical Steering Committee Symposium 6-8 July 1988*, Headquarters, United States Army Armament, Munitions and Chemical Command, Rock Island, Illinois, August 1991.

disperses rapidly downwind after reaching the cloud stabilization height (Figures 2.1 through 2.3). Typical propellant burns (Figures 2.4 and 2.5) produce extremely luminous, hot plumes over several seconds and with little soil involvement. They also permit more entrainment of ambient oxygen to assist in oxidation; however, they generate a less well-defined cloud, than surface detonations.

2.1.2.2. Because of the differences in explosive and propellant composition, geometry, and combustion phenomenology, it was suggested that chamber trials be conducted first using the following explosive and propellants:

2.1.2.2.1 The explosive 2,4,6-trinitrotoluene (TNT), the most oxygen-deficient (-74 percent) of the military explosives, and therefore the most likely to produce significant amounts of the products of incomplete combustion.

2.1.2.2.2 A double-base propellant containing primarily nitrocellulose and nitroglycerin with added ethyl cellulose.

2.1.2.2.3 A composite propellant containing ammonium perchlorate.

2.1.2.3. Product compositions and concentrations could then be compared with widely variable results reported in earlier computer-modeled combustion product studies and with laboratory-scale and less-controlled open-air detonation and burn studies reported in the literature.

2.1.2.4. To assess the effect that soil might have on product composition by its quenching of complete oxidation during TNT detonation, without puncturing the chamber's fabric walls, it was suggested that a surrogate fireball-mitigating material (aqueous foam) should be used to surround the TNT block on one of the detonation trials.

2.1.2.5. The air building chamber (the BB) at SNL (Figure 2.6) was chosen as the test facility because it offered several advantages over other facilities and devices designed to contain explosions. These advantages are as follows:

Figure 2.1 Typical Initial Fireball Formation, 907-kg (2000-lb) TNT OD Test.

Figure 2.2 Typical Cloud Formation With Entrained Soil, 907-kg (2000-lb) TNT Open Detonation Test.

Figure 2.3 Typical Stabilized Cloud Formation Prior to Downwind Dispersal, 907-kg (2000-lb) TNT Open Detonation Test.

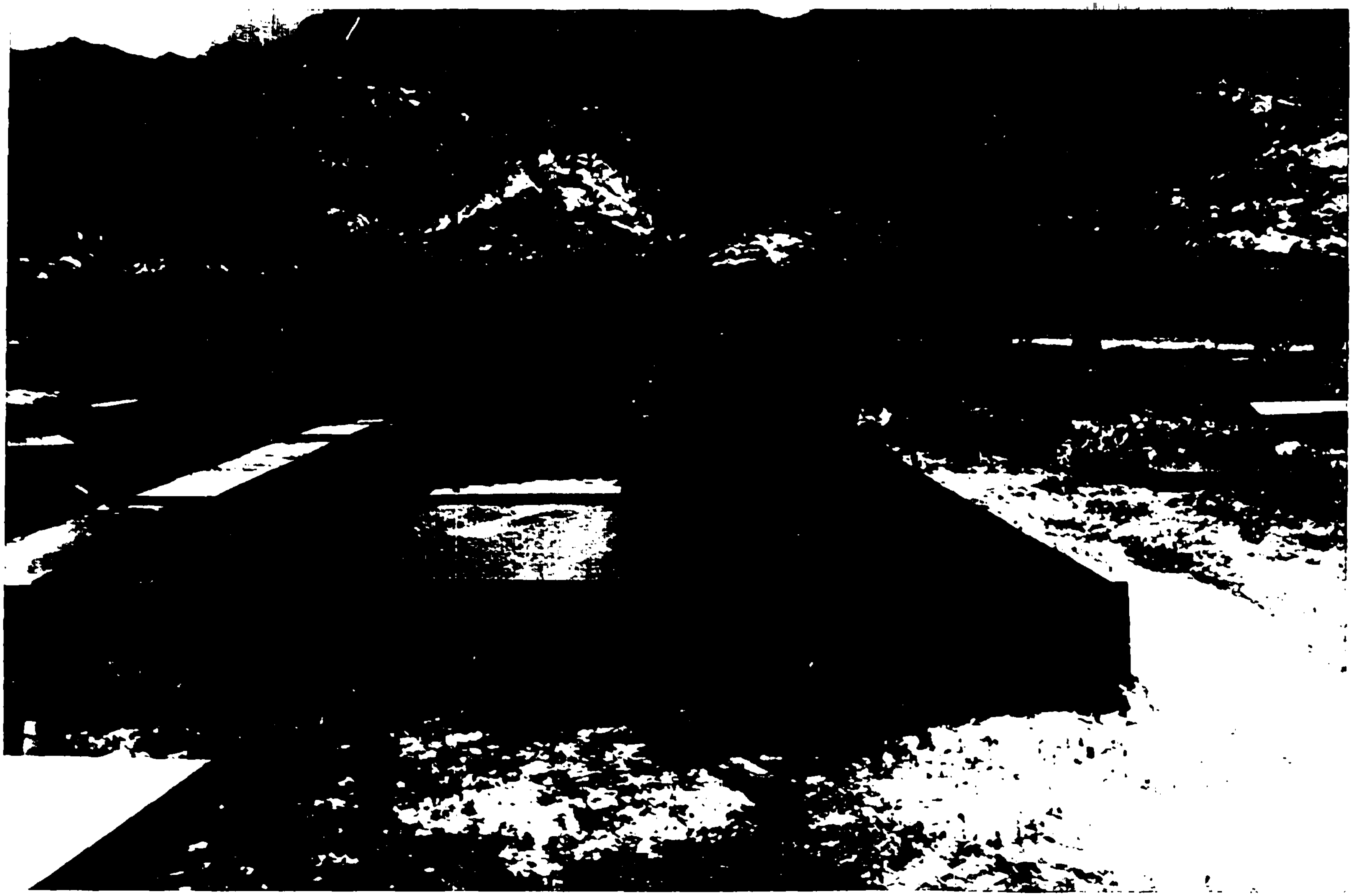

Figure 2.4 Partially-Filled Burn Pans, 3175-kg (7000-lb) Propellant Open Burn Test.

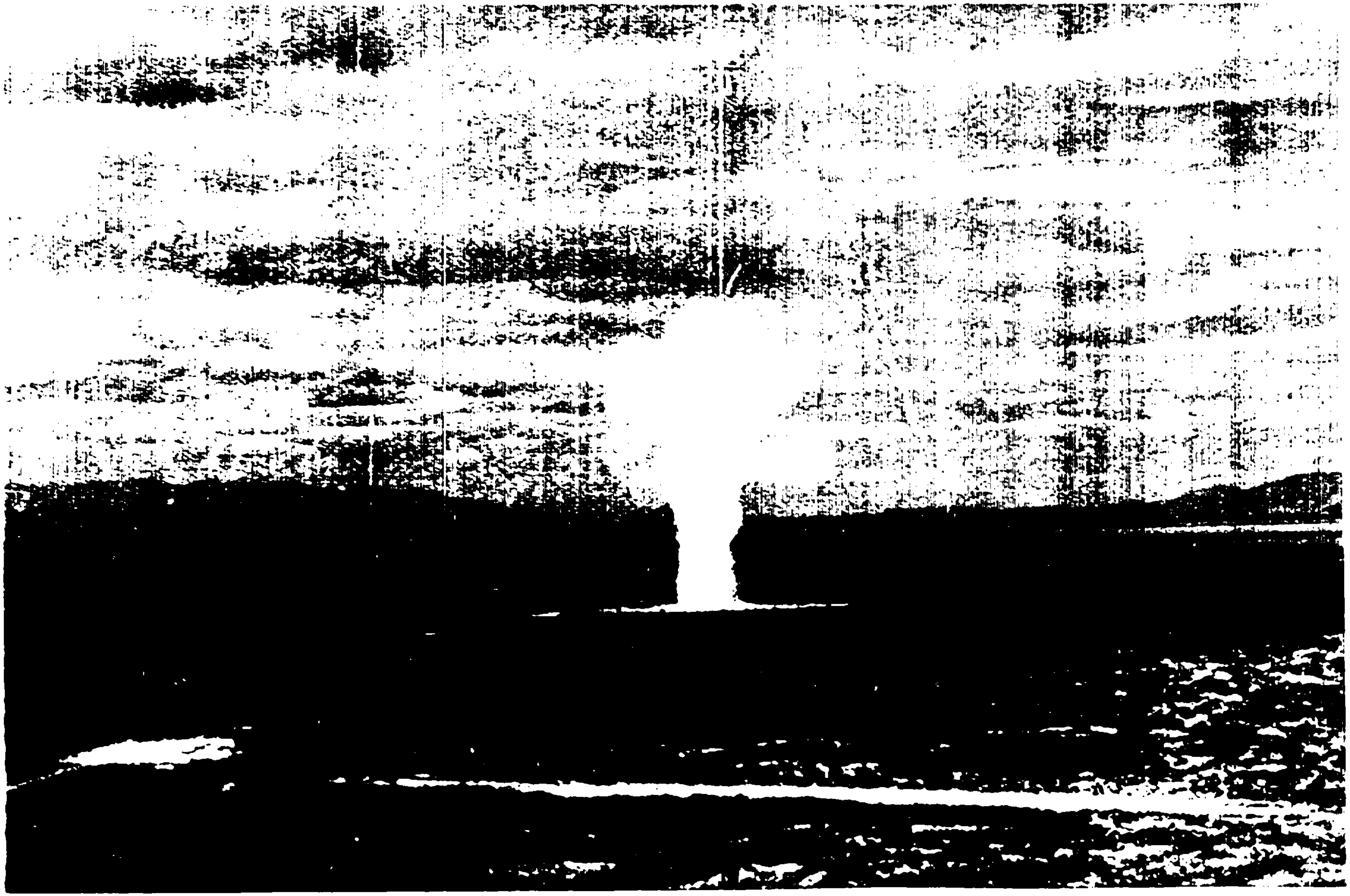

Figure 2.5 Typical Plume, 3175-kg (7000-lb) Propellant Open Burn Test.

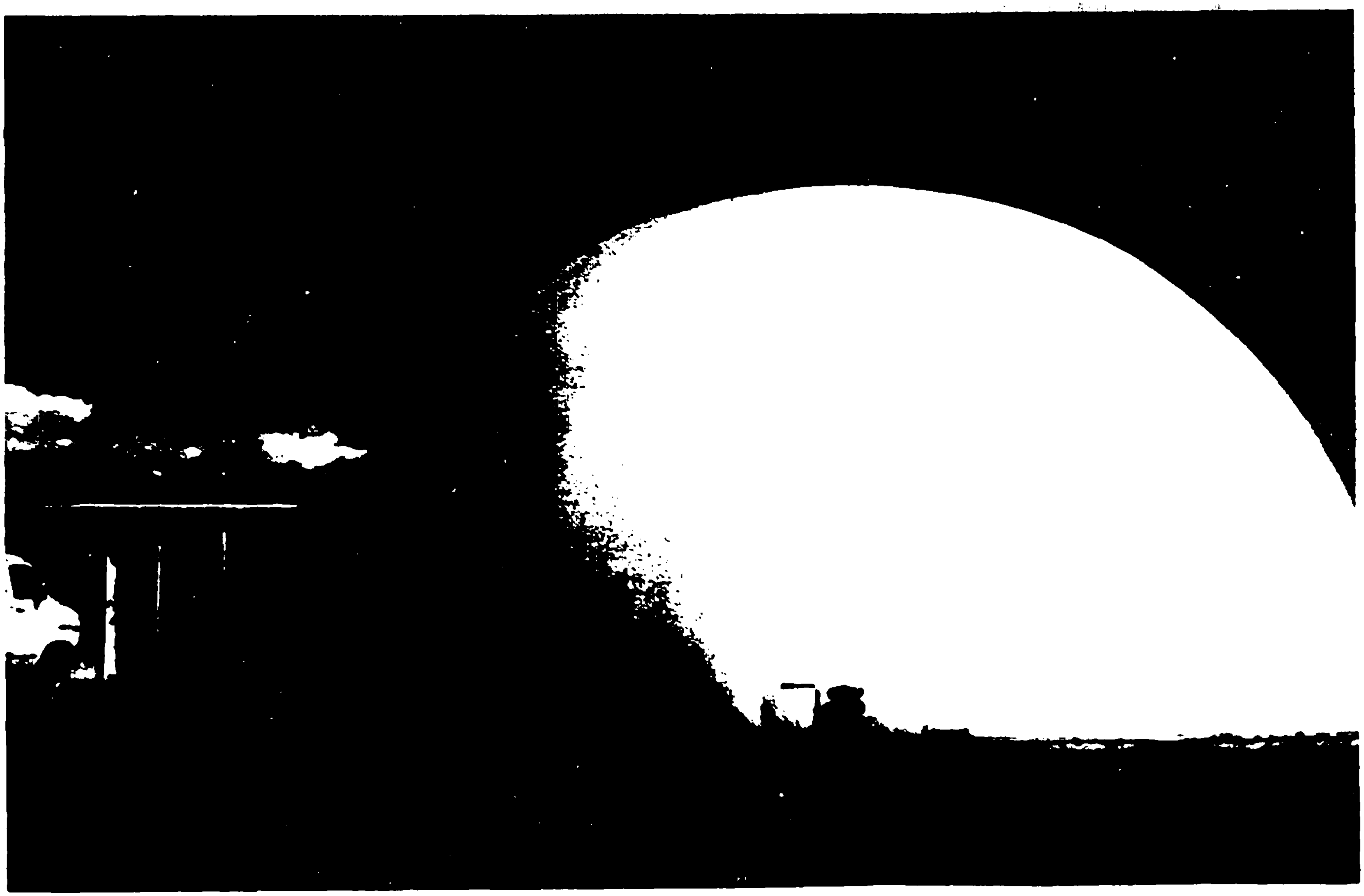

Figure 2.6 SNL BangBox Test Facility Containing Sampling and Real-Time Analysis Equipment.

2.1.2.5.1 It was large enough to permit complete combustion and containment of the product clouds from 227 grams (0.5 lb) of explosive and similar quantities of burning propellants.

2.1.2.5.2 There would be no significant spall from the aluminum-covered concrete floor to interfere with sample collection and analysis.

2.1.2.5.3 The same instruments, samplers, and data handling equipment that were proposed to be mounted in a fixed-wing aircraft (FWAC) to sample outdoor OB/OD clouds during later study phases could be completely checked under the controlled conditions of the BB trials. The FWAC planned for use is shown in Figures 2.7 and 2.8.

2.1.2.6. Characterization of the BB test facility included a determination of ventilation rate (using tracer gas) and a determination of the ability to achieve internal atmospheric homogeneity in the chamber using mixing fans. Sulfur hexafluoride (SF_6) and CO_2 would be used as tracer gases.

2.1.2.7. A list of target analytes would be developed covering gaseous criteria pollutants, volatile and semivolatile organic compounds, unreacted explosive/propellants and their manufacturing by products/contaminants, regulated metals and nonmetals, and other potentially detrimental organic compounds. The concentrations of these OB/OD products would then be determined by applying the most sensitive, reproducible, and versatile analytical methods available. Competing analytical technologies, such as gas chromatography-mass spectrometry (GC/MS) and supercritical fluid chromatography-mass spectrometry (SFC/MS), would be evaluated.

2.1.2.8. Sampling equipment and techniques would be evaluated for possible later use in the SNL FWAC during large-scale outdoor trials.

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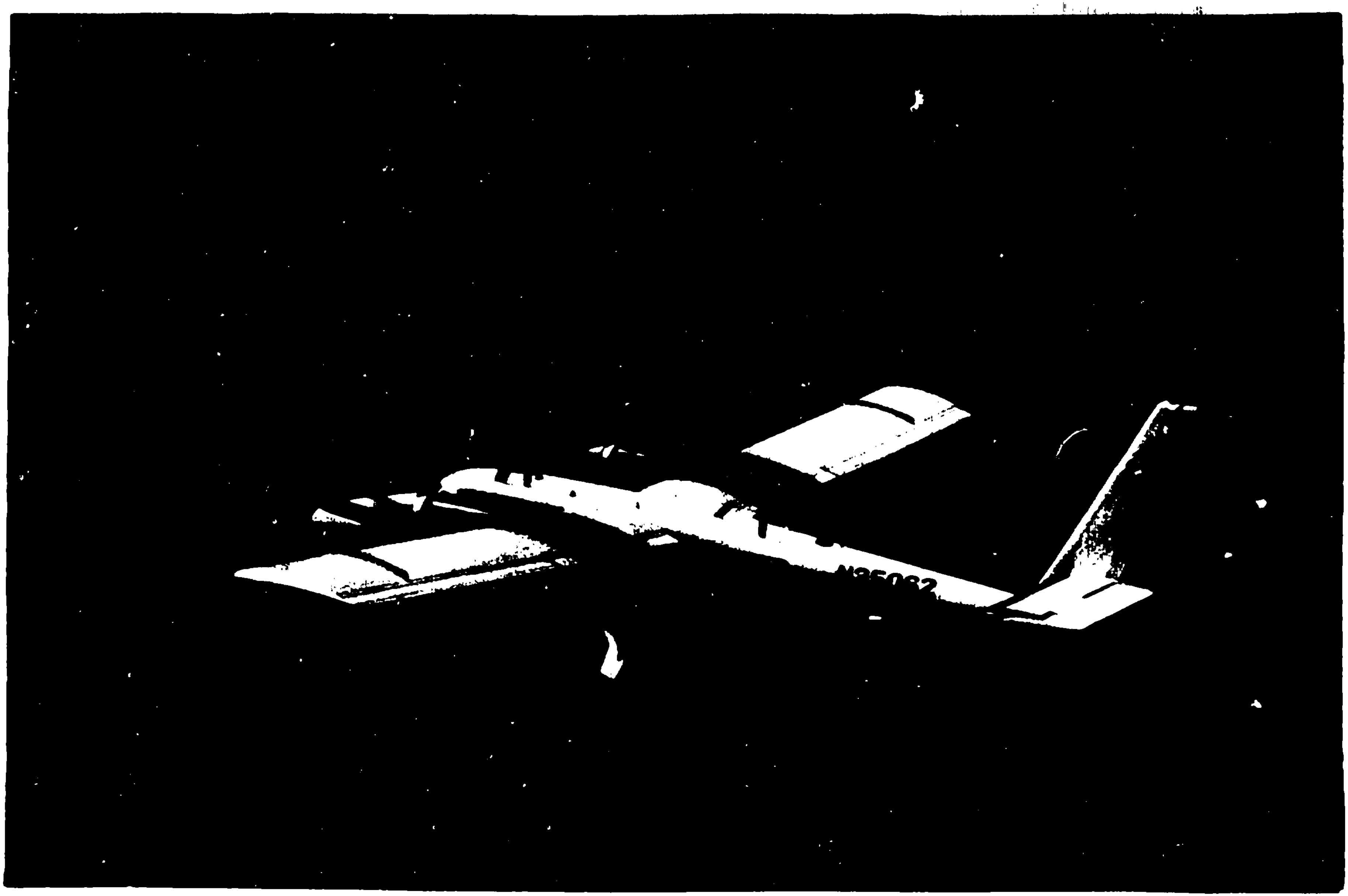

Figure 2.7 SNL Twin Otter Instrumented Fixed-Wing Aircraft for Open-Air OB/OD Sampling and Real-Time Gas Analysis.

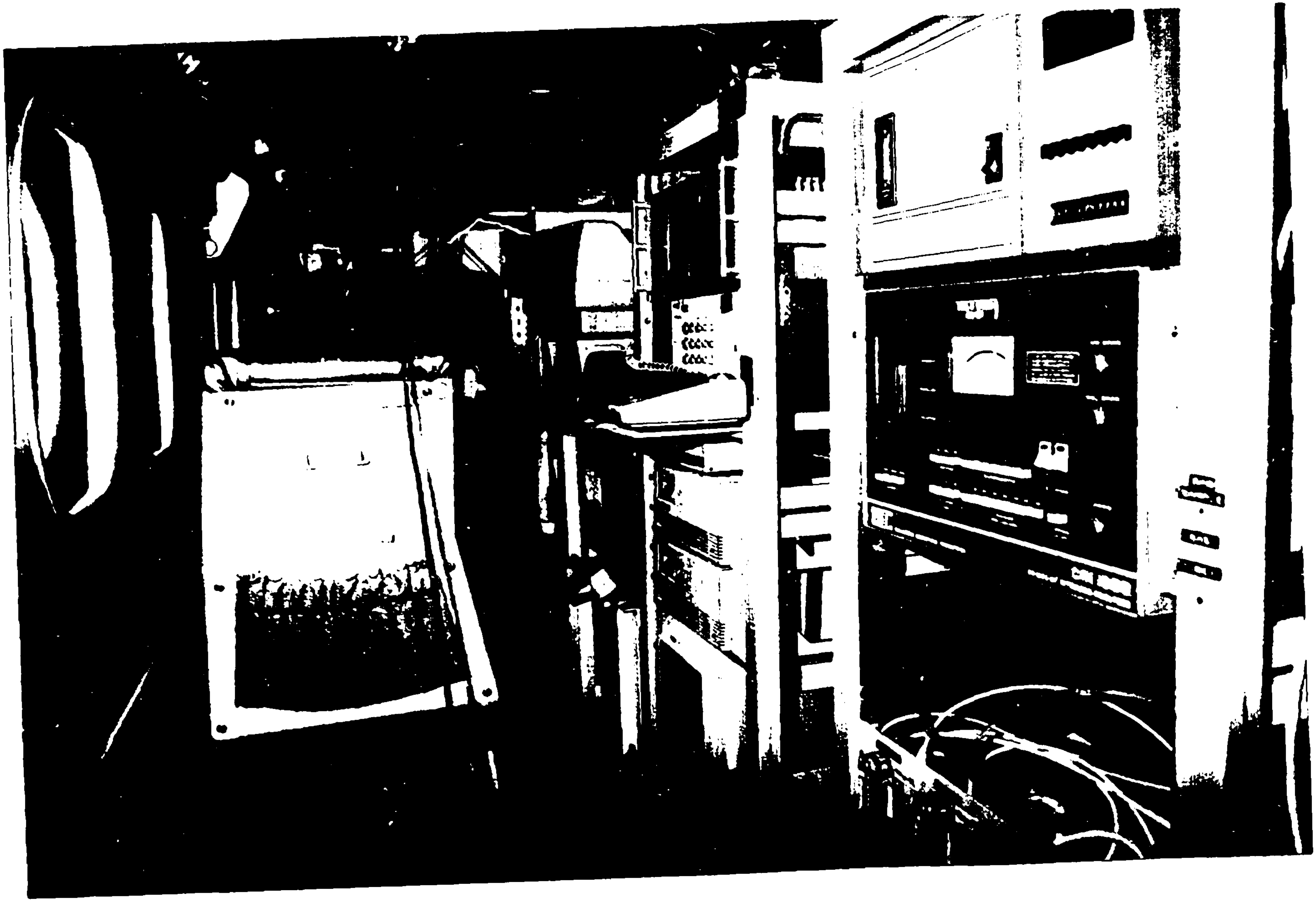

Figure 2.8 Interior View, Partial Instrument Array, SNL Fixed-Wing Aircraft.

2.1.2.9. The widely used method of estimating initial source strengths of emissions within OB/OD clouds yields extremely variable results, because the ultimate accuracy of the estimate depends not only on difficult-to-measure very low concentrations of compounds in the elevated, diffusing cloud, but also on very speculative estimates of the cloud's volume. SNL proposed an alternative method (the carbon balance method) to be evaluated during the chamber trials that did not depend on making a cloud volume estimate. This method would permit a more accurate calculation of emission factors of combustion products in diffusing clouds especially when the fuel's carbon content is well-characterized, and the concentrations of the major carbon-containing products can be accurately measured.

2.1.2.10. Adequate QA/QC procedures for use in sample collection, handling, analysis, and data treatment would need to be developed, checked, approved, and revised (where necessary) before conducting the expensive large-scale outdoor OB/OD trials.

2.2. Test Design Plan

After proposed solutions to technical issues were identified by the TSC, an AMCCOM test design plan was prepared. Formal BB testing began on 7 December 1988.

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SECTION 3. STUDY OBJECTIVES

3.1. Overall Test Program

3.1.1. Purpose

The broad overall program purpose is to supply waste characterization data for OB/OD permit applications under RCRA subpart X.

3.1.2. Objectives

3.1.2.1. Identify and/or develop sampling and analytical technology, instrumentation, and procedures needed to provide RCRA subpart X data characterization.

3.1.2.2. Identify and quantify emissions and residues produced by OB/OD thermal treatment methods.

3.1.2.3. Provide input for development and validation of an OB/OD dispersion model.

3.1.2.4. Identify specific items that can be treated by OB/OD thermal treatment methods without adverse environmental impact.

3.2. BangBox Test Series

3.2.1. Purpose

The OB/OD BB test series was designed to develop, verify and confirm the OB/OD thermal treatment method test technology/methodology.

3.2.2. Objectives

3.2.2.1. Characterize the BB chamber volume, ventilation rate, and combustion product cloud homogeneity level.

3.2.2.2. Develop and improve proposed air sampling equipment and sample analysis procedures to be used in later phases on the FWAC, for sampling product clouds from large-scale follow-on field OB/OD trials.

3.2.2.3. Refine, standardize, and compare supercritical-fluid chromatography (SFC) and gas chromatography (GC) techniques for extracting and analyzing resins, filters, and soils for trace quantities of semivolatile organic OB/OD combustion products and residues, using mass spectrometer (MS) detectors.

3.2.2.4. Verify adequacy of other standard analytical methods to be used for analyses of gases, particulates, volatile organic compounds, metals, and nonmetals.

3.2.2.5. Identify and quantify specific target analytes for TNT, a double-base propellant, and a composite propellant.

3.2.2.6. Assess polychlorinated dibenzodioxins (PCDD) and dibenzofurans (PCDF) levels generated from burning the composite propellant containing high concentration of NH_4ClO_4.

3.2.2.7. Provide information on the morphology, composition, and size distributions of airborne particulate material generated by OB/OD operations in the BB.

3.2.2.8. Examine, using data produced under controlled conditions, the validity of the proposed Carbon Balance method of calculating emission factors; compare the results with those calculated using the more-conventional cloud volume times concentration method.

3.2.2.9. Identify or develop appropriate program-specific QA/QC procedures.

3.2.2.10. Develop and establish procedures for transport and storage of sample specimens.

SECTION 4. TEST CONDUCT

4.1. Test Activities

Key test activities were conducted as outlined in Table 4.1.

Table 4.1 Key BangBox OB/OD Test Activities.

DATE	Key Test Activities
7 DEC 88	Single-charge (227-g) TNT OD: check out setup, equipment, procedures.
31 JAN 89	Single-charge (227-g) TNT OD data trial: chamber air homogeneity, volume, ventilation rate.
2 FEB 89	
6 FEB 89	
7 FEB 89	Extended (6-h) background air sampling inside and outside BB.
8 FEB 89	Multiple detonation (8 consecutive 227-g) TNT OD data trials: provided high concentrations of accumulated products.
9 FEB 89	454-g double-base propellant OB data trial.
13 FEB 89	Aqueous foam-attenuated (227-g) TNT OD data trial.
15 FEB 89	Multiple tank sampling 227-g TNT OD data trial: simultaneous air sample collection in 27 32-L tanks ("Big Gulp" trial).
16 FEB 89	454-g composite propellant OB data trial.

4.1.1. Facilities, Sampling Equipment, Materials, and Procedures.

4.1.1.1. As shown in Figure 2.6 (on page 2-13) the BB test facility is an air-supported, rubber-coated fabric hemisphere with a radius of 7.6 meters. Access to the building was through a plywood airlock, 5.5 x 2.1 x 2.5 meters in size. The building was supported by positive air pressure supplied by a blower. A damper on the outlet of the blower permitted adjustment of inflation airflow rate and positive pressure inside the building. The blower damper was manually adjusted to achieve an initial pressure differential of approximately 18 mm of water.

4.1.1.2. A number of OB/OD sampling instruments, normally installed on the FWAC, were positioned in the airlock and inside the chamber to test their performances. Gas and particulate samples were routed to airlock instruments via a 5-meter long, 8-cm diameter aluminum tube that projected 2 meters into the BB chamber. This tube is the same as that normally installed on the instrumented aircraft and serves as the sampling probe from which all particulate and gas samples are collected during flight. A matrix of instruments and equipment used in each trial is shown in Tables 4.2a and 4.2b.

4.1.1.3. The schematic layout of sampling equipment mounted in the BB airlock and used on trials subsequent to 7 December, 1989, is shown in Figure 4.1. The airflow from the interior chamber was routed through a pneumatically driven 10-cm diameter gate valve into a 1.5-m^3 carbon-impregnated polyethylene (Velostat™) sampling bag. The bag, which was constructed of electrically conductive plastic material to minimize wall loss effects of charged particles, fills with air from the chamber interior in approximately 40 seconds.

4.1.1.3.1 Stainless steel sampling lines led to a series of filters, vapor collection systems, and gas monitors. Particulate and semivolatile compounds were collected on two semivolatile organic sampling trains (SEMI-VOSTs), which consisted of a prefired quartz-fiber filter, followed by two sections containing XAD-2™ resin. The front and backup sections contained approximately 65 and 20 grams of resin, respectively. Other filters connected to the bag outlet manifold included a Teflon™ filter, used for gravimetric analysis and X-ray fluorescence (XRF) measurements, and a Nuclepore™ polycarbonate filter used for scanning electron microscopy (SEM). A pump provided airflow through the filters at a rate of approximately 200 L/min. Mass flowmeters enabled determination of air sample volumes.

4.1.1.3.2 Real-time continuous monitors used to provide data for this report are listed in Table 4.3. In-line Teflon™ filters were provided for all these monitors to prevent particulate contamination of the instrument optics. A differential mobility particle sizer (DMPS) was used to measure particle size distributions in the 0.01- to 0.5-μm diameter range. Grab samples of air were collected in electropolished stainless steel canisters directly from the sampling duct, and indirectly from the 1.5-m^3 sampling bag, by Oregon Graduate Center (OGC) personnel. The grab samples were analyzed by OGC for H_2, CO, CO_2 and C_1 - C_{10} hydrocarbon concentrations, using (GC) with thermal conductivity detector (TCD), flame ionization detector (FID) (for SF_6), and electron capture detector (ECD). <u>Hereafter, all samples collected and analyzed from the bag will be referred to as indirect samples.</u>

Table 4.2a Matrix of Instrument, Sampler, and Collector Used During the OB/OD Detonation BangBox Test.

	Trial Number[a]									
Item	1	2	3	4	5	6	7	8	9	10
PARTICLE SIZERS										
DMPS[b]	X	X	X		X	X	X	X		
APS[c]	X		X	X		X	X	X	X	X
FILTERS										
Teflon™			X	X			X	X		X
Nuclepore™			X	X			X	X		X
XAD-2™			X	X	X	X	X			X
Porapak-R™			X							
Quartz-fiber			X	X	X	X	X	X		X
DETECTORS										
Nephelometer			X	X		X	X	X	X	X
FID[d]			X					X		
PID[e]	X		X	X		X	X	X	X	X
ANALYZERS										
CO_2	X	X	X	X		X	X	X	X	X
CO	X	X	X	X		X	X	X	X	X
SO_2	X	X	X	X		X	X	X	X	X
O_3	X	X	X	X		X	X	X	X	X
NO_x	X	X	X	X		X	X	X	X	X
SF_6	X	X	X	X		X	X	X	X	X
BUBBLERS										
HCN			X	X		X	X			X
NH_3			X	X		X	X			X
HCl										X
BAG										
1-m^3	X		X	X			X	X		X

NOTE: Footnotes identified on next page.

Table 4.2b Matrix of Instrument, Sampler, and Collector Used During the OB/OD Detonation BangBox Test.

	Trial Number[a]									
Item	1	2	3	4	5	6	7	8	9	10
TANKS/CANISTERS										
0.85-L	X	X	X	X			X		X	X
6-L	X		X	X			X	X	X	X
32-L	X		X	X					X	
CAMERAS										
Color Video	X	X	X	X		X	X	X		X
HS[f]							X	X		X
HS Film	X		X							
MISCELLANEOUS										
Thermometer	X	X	X	X	X	X	X	X	X	X
Barometer	X	X	X		X	X	X	X	X	X
DP[g] Gauge	X	X	X	X		X	X	X	X	

[a] Trial: 1 - Homogeneity and BB Chamber Volume (1 Dec 88).
2 - Ventilation Rate (5 Dec 88).
3 - Single-Charge TNT OD (7 Dec 88).
4 - Single-Charge TNT ODs (31 Jan 89, 2 and 6 Feb 89).
5 - Extended Background Air Sampling (7 Feb 89).
6 - Multiple Detonation (8 Feb 89).
7 - Double-Base Propellant OB (9 Feb 89).
8 - Foam-Attenuated TNT Detonation (13 Feb 89).
9 - Multiple Tank Sampling ("Big Gulp") OD Trial (15 Feb 89)
10 - Composite Propellant Burn (16 Feb 89).

[b] Differential mobility particle sizer.
[c] Aerodynamic particle sizer
[d] Flame ionization detector.
[e] Photoionization detector.
[f] High-speed.
[g] Differential pressure.

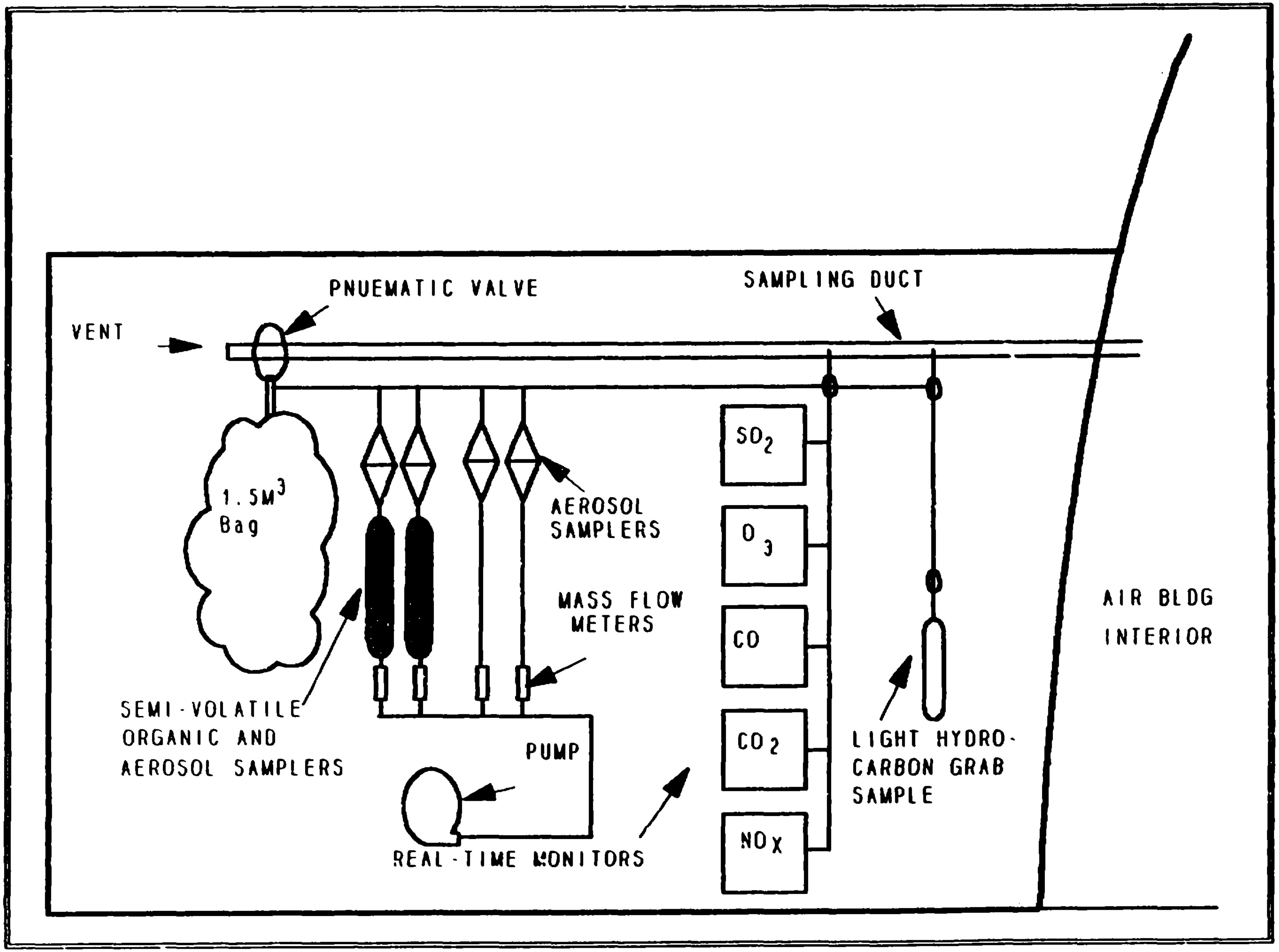

Figure 4.1 BangBox Airlock Instrumentation and Sampling Equipment.

Table 4.3 OB/OD Real-Time Continuous Monitors Positioned in the SNL BangBox Airlock.

SPECIES	INSTRUMENT	MEASUREMENT PRINCIPLE	DETECTION LEVEL[a]
CO_2	TECO Model 41H	Gas Filter Correlation	1.2 ppmv
CO	TECO Model 48	Gas Filter Correlation	0.1 ppmv
SO_2	TECO Model 43	Pulsed UV Fluorescence	2 ppbv
O_3	TECO Model 49	UV Absorption	5 ppbv
NO_x	CSI Model 1600	Chemiluminescence	6 ppbv
THC	Century OVA-128	Flame Ionization	2 ppmv
THC[b]	HNU Model PI-101	Photoionization	0.1 ppmv

[a] Detection level defined as two times the standard deviation of the instrument noise.

[b] The photoionization detector responds to other gaseous species in addition to hydrocarbons. No attempt is made in this study to correct for the non-specificity of this instrument. Hydrocarbons detected were quantitated separately by GC methods outlined elsewhere in this report.

4.1.1.3.3 In order to make direct measurements of interior chamber air following a detonation or burn, additional instruments and samplers were positioned inside the BB facility, as shown schematically in Figure 4.2. The various instruments included are listed in Table 4.4.

4.1.1.3.4 Two laser particle spectrometers that are normally installed in the SNL FWAC were used to make particulate measurements in real-time. Particles in the 0.15- to 3-μm diameter range were measured with an active scattering aerosol spectrometer probe (ASASP). Particles in the 2- to 47-μm diameter range were measured with a forward scattering spectrometer probe (FSSP). The FSSP probe is designed to incorporate true *in situ* measurement principles and, as such, requires no correction for particle transmission or sampling losses. Both the FSSP and ASASP probes provided records of total particle counts at 1-minute intervals.

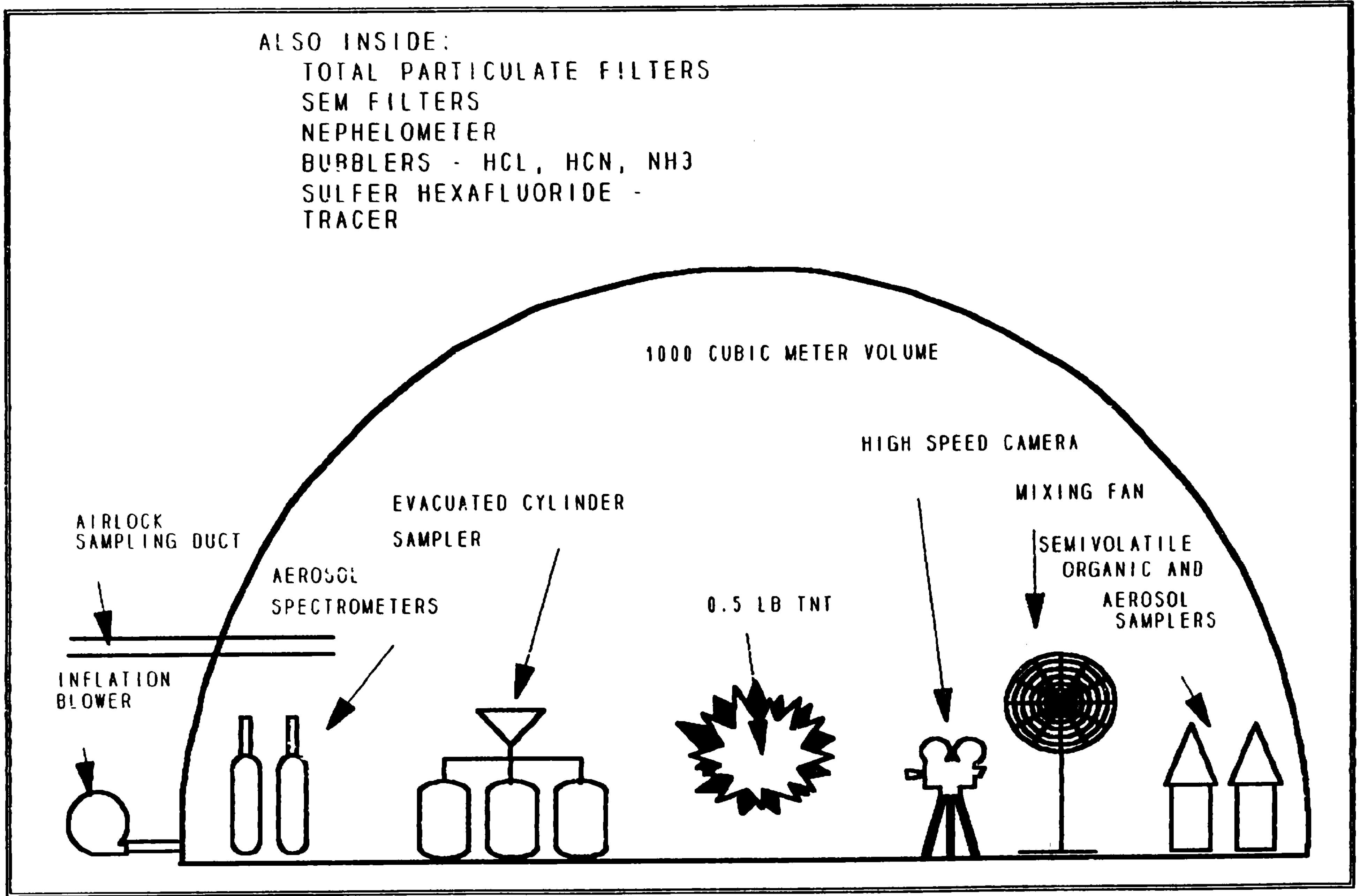

Figure 4.2 SNL BangBox Interior OB/OD Sampling Instrumentation and Equipment.

Table 4.4 Samplers Located Inside the BangBox.

Instrument - Parameter Measured	Model (Characteristics)
Aerosol spectrometer - particle size distribution	PMS ASASP-100X (0.15 - 3 μm diameter)
Aerosol spectrometer - particle size distribution	PMS FSSP-100-X (2 - 47 μm diameter)
Integrating nephelometer - particulate concentration	Belfort Ind. Model 1590 (0 - 3 μm diameter)
Nephelometer - particulate concentration	MIE Model RAM-1 (0 - 3 μm diameter)
Video camera - photometric record	NAC Model HSV-200 (200 frame/s)
Fast frame camera - photometric	NAC Model E-10 (500 - 10000 frame/s)
semi-VOST[a] (2) - trace organics	GMW Model PS-1 (≈ 100 L/min)
Teflon™ filter sampler - total particulate and metals.	GAST Model 30 (30 L/min)
Polycarbonate filter sampler - particle morphology	GAST Model 30 (3 L/min)
Bubbler sampler - Hydrogen cyanide	Gillian Model 113FS (2 L/min)
Bubbler sampler - Ammonia	Gillian Model 113FS (2 L/min)
Bubbler sampler - Hydrogen chloride[b]	Gillian Model 113FS (2 L/min)
Evacuated stainless steel canisters - volatile organics	OGC special design (6-L)
Evacuated stainless steel tanks - volatile and semivolatile organics	OGC special design (32-L)
Evacuated stainless steel canister - SF_6	OGC special design (0.85-L)

[a] semiVOST - semivolatile organic sampling train.
[b] Hydrogen chloride bubblers were used only on the composite propellant burn trial.

4.1.1.3.5 A flash-lamp integrating nephelometer and a portable forward light scattering particulate detector provided continuous measurement of particulate concentration inside the chamber during each test. Photographic coverage of detonation and burn trials was provided by video cameras and recorders and with a high-speed camera run at 5,000 frames per second. Two 1-meter diameter fans with approximate airflow rates of 250 m^3/minute were used to rapidly mix the BB chamber air prior to collection of "homogeneous" air samples. Samples collected from the chamber interior immediately after detonation or burn and before turning on the mixing fans are considered to be nonhomogeneous.

4.1.1.3.6 Semivolatile and organic particulate species were collected directly from the chamber interior by two modified semiVOSTs operating at flow rates of approximately 100 L/min. The filter and cartridge units used in these semiVOSTs were identical to those used for collection of air drawn from the 1.5-m^3 sampling Velostat bag mounted in the airlock. Standard high-volume sampler blowers were used to pull air through these direct semiVOST units.

4.1.1.3.7 As a result of these sampling arrays, data from both direct (chamber) and indirect (bag) sampling during both nonhomogeneous and homogeneous air sampling periods could be compared. Glass impingers (bubblers) filled with appropriate absorbing solutions were used to collect ammonia and hydrogen cyanide product gases.

4.1.1.3.8 An audit of criteria gas and mass flow instrument performance was conducted by personnel from the U.S. Environmental Protection Agency (EPA), Atmospheric Research and Exposure Assessment Laboratory, Quality Assurance Division, Research and Monitoring Evaluation Branch, Research Triangle Park (RTP), North Carolina. Results of the audit were excellent.

4.1.1.3.9 Teflon™ filter particulate sample weights were determined gravimetrically by precision electrobalance. The Teflon™ filters were then analyzed by XRF spectroscopy (for elements with atomic mass units in excess of 12) by Lawrence Berkeley Laboratory (LBL). Particulate carbon speciation was done by Sunset Laboratory (SSL) on 1-cm^2 punches taken from SEMI-VOST quartz-fiber filter samples. This thermo-optical analysis involves a two-step volatilization and combustion process to differentiate between organic, elemental, and inorganic carbon on the filter. Bubbler samples were analyzed by National Institute for Occupational Safety and Health (NIOSH) method 205 (colorimetric) for NH_3 and NIOSH method 116 (cyanide-specific ion electrode) for HCN. Trace-level semivolatile organic compounds from semiVOST filters, XAD-2™ resins, cartridges, swab samples, and 32-L tanks were identified, separated, and quantified by both Battelle Columbus Division (BCD) and by Alpine West Laboratories (AWL). Battelle used GC/MS and AWL used SFC/MS for analysis of solutions prepared by extraction of sample media with methylene chloride followed by concentrating by rotary evaporation.

4.1.1.3.10 During the composite propellant burn, high-volume air samplers sampling at 283 L/min were also used to take duplicate samples on quartz-fiber filters. The filters were backed up by pre-cleaned polyurethane foam-filled cartridges. Analyses were made for PCDD and for PCDF by BCD. The analyses included determination of total hepta-, hexa-, penta-, and tetra-chlorinated dibenzodioxin (CDD) and -chlorinated dibenzofuran (CDF) congeners, as well as octa- and 2,3,7,8-tetra-CDD and - CDF concentrations. Taking into account the relative toxicities of the congeners and converting the results to an equivalent quantity of the most toxic, only 2 ng of PCDD and 3.3 μg of PCDF were detected per ton of composite propellant.

4.2. Chronology

A typical chronology of events for a TNT detonation trial is shown in Table 4.5. The chamber was washed and the floor vacuumed before detonation trials began. Event chronology for the propellant burn and foam-attenuated TNT detonation trials was similar, except for fuel setup configurations and modifications of sampling procedures because of unique data requirements for those trials. A typical detonation of a suspended rectangular 227gram (0.5 lb) TNT block is shown in Figure 4.3. On completion of each trial, samples were collected, sealed, stored at ambient temperatures (usually 0 to 5°C), and subsequently transported to the various laboratories for extraction.

Table 4.5 Typical Chronology of Events for a TNT Detonation Trial.

Time[a] (min)	Event
t-75	Install background filters, SEMI-VOST cartridges, bubblers. Record initial flow rates on all samplers. Check zero and span on all continuous monitors.
t-60	Clear all personnel from chamber interior. Begin background sampling with all instruments and samplers.
t-30	Complete background sampling. Record final flow rates on all samplers. Remove background sampling media. Install all test filters, SEMI-VOST cartridges, bubblers, etc. Record initial flow rates on all samplers.
t-15	Hang 227-g (0.5-lb) TNT charge in test fixture.
t-5	Start data acquisition on all continuous instruments. Clear all personnel to safe bunker. Connect detonator cable to charge and arm.
t-0	Detonate charge.
t+3	Remotely start all filter and SEMI-VOST samplers inside chamber. Collect nonhomogeneous sample No. 1 from sampling bag. Pump bag sample through sampling media in airlock. Switch criteria gas monitors to sample directly from bag for 3 minutes. Fill 6-L evacuated cylinder from interior of chamber.Fill 6-L evacuated cylinder from bag.
t+5	Switch mixing fans on for 3 minutes.
t+10	Change sampling media on bag system.
t+15	Collect homogeneous (mixed) air sample No. 1 in sampling bag. Pump bag sample through sampling media. Switch criteria gas monitors to sample directly from bag for 3 minutes. Fill 6-L evacuated cylinder from interior of chamber. Fill 6-L evacuated cylinder from bag.
t+25	Change sampling media on bag system.
t+30	Collect homogeneous sample No. 2 from sampling bag. Pump bag sample through sampling media. Switch criteria gas monitors to sample directly from bag for 3 minutes. Fill 6-L evacuated cylinder from chamber interior. Fill 6-L evacuated cylinder from bag.
t+35	Stop direct SEMI-VOST, filter, and bubbler samplers. Reenter chamber and measure final sampler flows.
t+45	Check zero and span on all continuous monitors. Retrieve and back-up all test data. Collect and preserve all sampling media.

[a]t = time of detonation.

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Figure 4.3 Detonation Fireball from Suspended 227-g (0.5-lb) TNT Block Detonation in BangBox.

SECTION 5. SAMPLE ANALYSIS AND ANALYSIS RESULTS

5.1. Sample Handling and Analysis

Sample handling and analysis procedures are outlined in individual laboratory Letters of Instruction (LOIs) (See Volume 3).

5.2. Chemical Compounds Targeted for Analysis

5.2.1. Potentially hazardous substances for which present data was most lacking relative to OB/OD operations were the trace organic compounds, sometimes called products of incomplete combustion (PIC), formed in any combustion process that is less than ideal. The application of the term PIC to OB/OD operations is somewhat misleading, because it may imply that these organic substances occur as a part of the original mass of uncombusted material that happens to escape complete oxidation. In actuality, the OB/OD PIC are formed by pyrolysis of organic matter at flame temperatures. They occur in the combustion effluent because insufficient oxygen is present and/or thermodynamic equilibrium with the environment is not achieved because of premature quenching. Because of electron delocalization, these exotic organic compounds have large atomic bonding energies, and are thermodynamically stable at high temperatures. Furthermore, certain polycyclic aromatic hydrocarbons, along with their nitrogen or oxygen heterocyclic counterparts and their nitro and/or amino derivatives, are among the most mutagenic of known organic compounds. Consequently, such compounds received major attention when targeting certain semivolatile organic compounds for specific analysis. The PIC of greatest interest to OB/OD activities can be placed in eight main groups: (1) residual primary explosives, (2) propellants and their contaminants; (3) inorganic gases, (4) particulates, (5) volatile and semivolatile (exotic) organics, (6) other pyrolysis/combustion products, (7) metals, and (8) anions. Target compounds for detailed analysis from each of these groups were selected by the technical steering committee in July 1988; the target analyte lists were modified slightly after preliminary results were studied.

5.2.2. A consolidated list of chemical compounds targeted for analysis (target analytes), together with a notation as to whether or not they were detected above background levels by detailed sample analysis, is shown in Tables 5.1a, b, c, d, e, and f.

Table 5.1a Target Analytes Detected Above Background Levels for TNT Detonations, Double-Base Propellant Burn, and Composite Propellant Burn in the BangBox Test.

Species	TNT	Double-Base	Composite
Carbon dioxide	X[a]	X	X
Carbon monoxide	X	X	X
Nitrogen dioxide	X	X	X
Nitric oxide	X	X	X
Sulfur dioxide	X	X	X
Ozone	-[b]	X	ND[c]
Methane	X	X	X
Acetylene	X	X	X
Benzene	X	X	X
PARAFFINS			
n-Heptane	X	X	X
2,4-Dimethylhexane			
2-Methylheptane	X	X	X
2-Methylpentane	X	X	
3-Methylpentane	X	X	
Ethylcyclohexane			
n-Hexane	X	X	X
i-Butane	X	X	
Methylcyclopentane	X	X	X
n-Butane	X		
2,4-Dimethylpentane	X		
2,2-Dimethylpropane			
Cyclohexane	X	X	
n-Pentane	X	X	
2,3-Dimethylpentane	X	X	X
Cyclopentane	X	X	X
3-Methylhexane	X		X
n-Octane	X	X	X
Ethane	X	X	X
2,3-Dimethylhexane	X		
Methylcyclohexane	X	X	X
2,3,4-Trimethylpentane	X	X	
n-Nonane	X	X	X

See Table 5.1f for notes.

Table 5.1b Target Analytes Detected Above Background Levels for TNT Detonations, Double-Base Propellant Burn, and Composite Propellant Burn in the BangBox Test.

Species	TNT	Double-Base	Composite
PARAFFINS (cont'd)			
2,3-Dimethylbutane	X	X	X
2,2,3-Trimethylpentane			
i-Pentane	X	X	X
Propane	X	X	X
2,2-Dimethylbutane	X		X
3-Ethylhexane	X	X	X
OLEFINS			
Ethylene	X	X	X
2-Methyl-1-pentene			
Propene	X	X	X
1-Butene	X	X	X
trans-2-Hexene			
3-Methyl-1-butene			
Mycrene			
Isoprene			
1,3-Butadiene	X	X	X
trans-2-Pentene			
cis-2-Butene		X	X
cis-2-Hexene			X
1-Pentene	X		
2-Methyl-2-butene			
1-Hexene			
4-Methyl-1-pentene			X
trans-2-Butene		X	X
i-Butene	X	X	X
2-Methyl-2-pentene			
2-Methyl-1-butene	X	X	
cis-2-Pentene			
Cyclopentene		X	X

See Table 5.1f for notes.

Table 5.1c **Target Analytes Detected Above Background Levels for TNT Detonations, Double-Base Propellant Burn, and Composite Propellant Burn in the BangBox Test.**

Species	TNT	Double-Base	Composite
NONBENZENE AROMATICS			
Toluene	X	X	
3-Ethyltoluene	X	X	
1,3,5-Trimethylbenzene			
n-Propylbenzene		X	
1-Ethyltoluene			
Styrene	X	X	X
i-Propylbenzene			
2-Ethyltoluene			
Ethylbenzene	X	X	X
o-Xylene	X	X	X
p-Xylene	X	X	X
m-Xylene			
1,2,4-Trimethylbenzene & sec-Butylbenzene			
TERPENES			
β-Pinene			
α-Terpinene			
d-Limonene			
α-Pinene			
Δ^3-Carene			
γ-Terpinene			
Terpinolene			
SEMIVOLATILE ORGANICS			
2,6-Dinitrotoluene	X	X	X
4-Nitrophenol	X	X	X
2,4-Dinitrotoluene	X		X
2-Nitronaphthalene	X	X	X
2,4,6-Trinitrotoluene	X	X	
1-Nitropyrene	X		X
1,6-Dinitropyrene			
Phenol	X	X	X
Dibenzofuran	X	X	X

See Table 5.1f for notes.

Table 5.1d Target Analytes Detected Above Background Levels for TNT Detonations, Double-Base Propellant Burn, and Composite Propellant Burn in the BangBox Test.

Species	TNT	Double-Base	Composite
SEMIVOLATILE ORGANICS (cont'd)			
2-Naphthalenamine			
Diphenylamine			
Benz[c]acridine			
Benz[a]anthracene	X		
Benzo[a]pyrene	X	X	
Dibenz[a,h]anthracene	X		
N-Nitrosodiphenylamine	X	X	X
Naphthalene	X		X
1-& 2-Methylnaphthalene	X	NA[d]	NA
1,3,5-Trinitrobenzene	X	NA	NA
Biphenyl	X	NA	NA
Phenanthrene	X	NA	NA
2,5-Diphenyloxazole	X	NA	NA
1,1,3-Trimethyl-3-phenylindane	X	NA	NA
Pyrene	X	NA	NA
4-Nitrosodiphenylamine	NA		NA
2-Nitrodiphenylamine	NA		NA
4-Nitrodiphenylamine	NA		NA
Nitroglycerin	NA		NA
Resorcinol	NA		NA
Di-n-propyl adipate	NA		NA
Triacetin	NA		NA
Salicylic acid	NA		NA
2,2-Methylene bis(4-methyl)-t-butylphenol	NA	NA	
Phenyl di-isodecyl phosphite	NA	NA	
5-ethyl-1,3-diglycidyl-5-methyl hydantoin diepoxide	NA	NA	
Diethylenetriamine	NA	NA	
Dioctyl sebacate	NA	NA	
Isophorone di-isocyanate	NA	NA	

See Table 5.1f for notes.

Table 5.1e Target Analytes Detected Above Background Levels for TNT Detonations, Double-Base Propellant Burn, and Composite Propellant Burn in the BangBox Test.

Species	TNT	Double-Base	Composite
METALS			
Aluminum	X		X
Silicon	X		X
Sulfur	X		X
Chlorine	X		X
Potassium	X		X
Calcium	X		X
Titanium			
Vanadium			X
Chromium	X		X
Magnesium	X		X
Iron	X		X
Nickel	X		X
Copper	X	X	X
Zinc	X		X
Gallium	X		X
Germanium	X		
Arsenic			
Selenium	X		
Bromine	X		
Uranium	X		
Strontium	X		
Lead	X	X	X
Zirconium	X		
Molybdenum	X		
Silver	X		
Cadmium	X		
Tin	X		X
Antimony	X		X
Iodine	X		
Barium	X		X

See Table 5.1f for notes.

Table 5.1f Target Analytes Detected Above Background Levels for TNT Detonations, Double-Base Propellant Burn, and Composite Propellant Burn in the BangBox Test.

Species	TNT	Double-Base	Composite
BUBBLERS			
Ammonia	X		
Hydrogen Cyanide[e]			
Hydrogen Chloride			X

BLANK SPACE - analyzed - not detected above background levels.
[a]X-detected above background levels.
[b]Ambient ozone reduced to below background levels immediately after the detonation.
[c]ND denotes no data.
[d]NA denotes not targeted for analysis for that type of trial.
[e]Analysis of bubbler samples enabled estimation of the CN- anion (as HCN).

5.3. Emission Factor Calculations

5.3.1. Parameter Values

Before EFs for each BB OB/OD product could be calculated for the concentration times volume method and the carbon balance method, several important parameter values were needed: BB volume, BB ventilation rate, total quantity of carbon in the fuel (TNT and propellants), total (combined) carbon content of the compounds generated from the detonation or burn, and the concentrations of the compounds in the samples collected.

5.3.2. BangBox Volume and Ventilation Rates

Volume of the BB for each trial was determined by analyzing data from release of known amounts of SF_6, a tracer gas whose concentration was then carefully measured. These volumes varied (from trial to trial) between 759 and 1078 m^3. The appropriate value, pertinent to the specific trial day, was used in analyzing each trial's data. Trial-specific BB ventilation rates were also determined and served as the bases for determining the "time zero" values for target analyte air concentrations; i.e., the initial postdetonation source strengths of each product. These ventilation rates were extremely well-characterized because of the high quality (accuracy and precision) of the SF_6 data collected. A typical plot of instrument voltage versus time for the CO_2 monitor (Figure 5.1) shows that homogeneity is achieved after 3 minutes of mixing with fans (generally fans are turned on 3 to 5 minutes after detonation) and then the chamber air containing combustion products is slowly diluted (ventilation) as makeup air is brought into the chamber to keep it inflated. Typical plots of net CO_2 concentration above background levels (including 95-percent confidence limits), as measured by real-time instruments during the homogeneous period of sampling after one of the 8 February 1989 227-gram TNT detonations, are shown in Figure 5.2.

5.3.3. Concentrations of Target Analytes in Air Samples

5.3.3.1. Target analyte concentrations in the bangbox air immediately after OB or OD were determined by the variety of sensitive analytical methods listed in Table 5.2. The most unique of these methods was application of the relatively new technique of supercritical fluid chromatography (SFC) for separation of semivolatile organic compounds in extract solutions of filter- and canister-collected samples of BB air. SFC was chosen for use in the OB/OD program, because certain thermally unstable OB/OD products that are converted to other compounds *during analysis* by GC remain stable when separated by SFC. An example is N-nitrosodiphenylamine, which is specifically identifiable by SFC but converts to diphenylamine when subjected to GC. However, certain volatile compounds, such as phenol, are not easily detected by SFC when supercritical CO_2 is used; these compounds *are* easily detected by GC. Other detection methods such as HPLC, were not considered sensitive enough for the scope and detection limits needed.

5.3.3.2. The extremely sensitive methods of chemical ionization negative and positive selective ion monitoring by mass spectrometry were used for product quantification with both SFC and GC separation techniques. Semivolatile organic compound assay results by SFC/MS and GC/MS showed that *both* separation techniques are applicable and that MS detection and quantification are extremely sensitive. For example, the laboratory responsible for the SFC/MS analyses reported typical lower detection limits (signal-to-noise >3) for compounds in methylene chloride solution of 0.03 to 2.3 ng/mL, depending on the specific analyte being assayed.

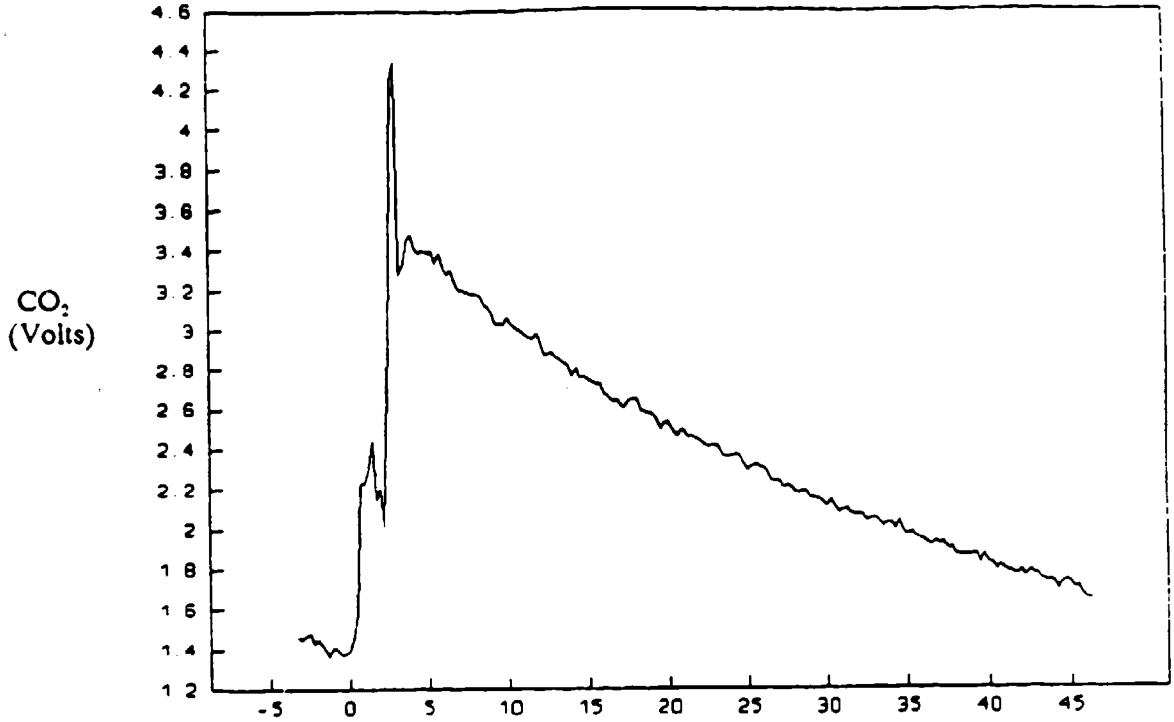

Figure 5.1 CO$_2$ Instrument Voltage Trace, TNT Detonation Number 2, 8 February 1989.

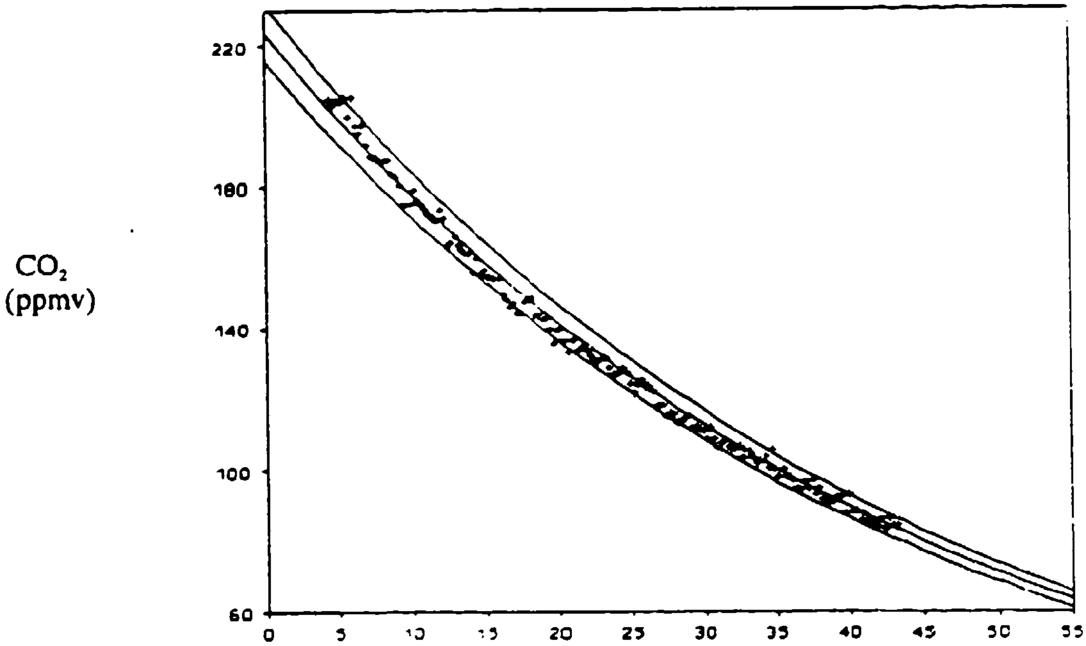

Figure 5.2 CO$_2$ Concentration and 95 Percent Confidence Limits During the Homogeneous Period of Direct Sampling From the BangBox TNT Detonation Number 2, 8 February 1989.

Table 5.2 Summary of OB/OD BangBox Sample Analysis Program.

TYPE	TARGET SPECIES	ASSAY METHOD	ASSAY LAB
Exotic organics	Approx 33 compounds	SFC-MS[a]	AWL[b]
Exotic organics	Approx 15 compounds	GC-MS[c]	BCD[d]
Inorganic (metals)	7	XRF[e]	LBL[f]
Total particulate carbon	Organic, elemental, and inorganic carbon	Thermooptical	SL[g]
Volatiles	THC[h], CH_4, H_2, C_2-C_{10}, C_6H_6, CO, CO_2	GC[i]	OGC[j]
Particle characterization	NA[k]	SEM[l]	SNL[m]
Near-real-time	SF_6	GC/ECD[n]	OGC
Real-time	CO, CO_2, NO_x[o], SO_2, O_3, THC	Gas and total hydrocarbon analyzers	SNL
Bubbler	HCN, HCl, NH_3	NIOSH standard methods	SNL
Particle size/mass	Particulate	DMPS[p], APS[q], ASASP[r], FSSP[s]	SNL
Dibenzodioxin compounds Dibenzofuran compounds	Specified	GC-MS	BCD

[a] Supercritical fluid chromatography-mass spectrometry.

[b] Alpine West Laboratory.

[c] Gas chromatography-mass spectrometry.

[d] Battelle Columbus Division.

[e] X-ray fluorescence.

[f] Lawrence Berkeley Laboratory.

[g] Sunset Laboratory.

[h] Total hydrocarbons.

[i] Gas chromatography.

[j] Oregon Graduate Center.

[k] Not applicable.

[l] Scanning electron microscopy.

[m] Sandia National Laboratories.

[n] Electron capture detector.

[o] Nitrogen oxides.

[p] Differential mobility particle sizer.

[q] Aerodynamic particle sizer.

[r] Active scattering aerosol spectrometer probe.

[s] Forward scattering spectrometer probe.

5.3.4. Audit samples

Audit samples in the form of spiked canisters were provided by the EPA, RTP NC. These samples were successfully analyzed (as ascertained by EPA auditors) by laboratories involved in both volatile and semivolatile organic compound analyses (AWL, BCD, and OGC).

5.4. Carbon Mass Balance.

5.4.1. Analysis

Analysis of the aerosol and particulate samples taken during a given trial provided an estimate of carbon mass released, based on the combined carbon mass contained in generated CO_2 and CO, the organic particulate/semivolatile carbon, volatile organic compound (C_1-C_{10} hydrocarbons), elemental carbon (soot), HCN, and inorganic carbon (as carbonate). The carbon from CO_2 was estimated from the real-time continuous monitor, with extrapolation of the fitted exponential curve (fit to the data from the homogeneous period of sampling) to detonation time zero ($t = 0$) . As noted earlier, the carbon from CO and volatile C_1-C_{10} hydrocarbons was estimated from the results of 6-L canister sampling, with extrapolation of the fitted exponential curve to detonation time zero. Total particulate organic carbon, elemental carbon (soot), and inorganic carbonate were estimated from thermal analysis of 1-cm^2 samples taken from quartz-fiber filters. This latter analysis incorporates a two-step volatilization and combustion process that permits estimation of the contributions by each of these carbon sources.

5.4.2. Carbon Mass

Table 5.3 shows the carbon mass derived from each source during 11 TNT detonations, as well as ratios of total carbon detected to that predicted as available from the TNT fuel (37.01 percent). The carbon mass determined by the analysis of sampled aerosol and particulate carbon sources overaccounts for the (theoretical) mass of carbon available in bulk TNT by about 9 percent. Results showed that the total carbon amounts contributed by volatile organics (C_1-C_{10}), HCN, and inorganic carbonate are negligible (<0.01 percent), while CO_2 contributes the vast majority of carbon measured (97.2 percent). Minor amounts of CO (0.50 percent) carbon, carbon from semivolatile principally to CO_2, upon detonation in ambient air. Table 5.3 also shows the carbon mass derived from the foam-attenuated TNT detonation, the double-base propellant burn and the composite propellant burn. The overestimate of the carbon mass (0.29) in the composite propellant burn could easily be the result of uncertainty in the carbon content of the composite propellant. Three composite propellant samples assayed for elemental carbon gave percentages of carbon ranging from 12.67 to 21.26.

5.4.2.1. These results are in sharp contrast to those predicted by certain theoretical models or determined during experiments involving limited available oxygen from surrounding air. The results

Table 5.3 Measured Carbon Mass Derived From Each Source Resulting From TNT Detonation or Propellant Burn.

DATE	SOURCE	CARBON MASS MEASURED (g)				Total Carbon (g)		RATIO[d]
		CO_2	CO	OC[a]	EC[b]	MEASURED[c]	THEORETICAL	
31 JAN 89	TNT	94.96	0.6688	0.4161	1.0331	97.08	82.90	1.17
2 FEB 89		82.18	0.4093	0.6495	1.5160	84.75	82.31	1.03
6 FEB 89		82.92	0.3384	0.4685	2.0492	85.78	83.22	1.03
8 FEB 89-2[e]		90.34	0.4578	0.5150	1.5426	92.86	82.99	1.12
8 FEB 89-3		90.26	0.4578	0.5150	1.5426	92.77	83.25	1.11
8 FEB 89-4		87.43	0.4578	0.5150	1.5426	89.95	82.62	1.09
8 FEB 89-5		87.15	0.4578	0.5150	1.5426	89.66	82.71	1.08
8 FEB 89-6		87.92	0.4578	0.5150	1.5426	90.43	82.42	1.10
8 FEB 89-7		86.74	0.4578	0.5150	1.5426	89.26	82.47	1.08
8 FEB 89-8		85.86	0.4578	0.5150	1.5426	88.37	81.87	1.08
15 FEB 89		83.78	0.3813	0.4751	1.4229	86.06	81.88	1.05
							Average	1.09
							Std. Dev.	0.04
13 FEB 89	TNT w/foam	24.13	2.7154	NS[f]	NS	26.85	82.00	0.33
9 FEB 89	Double base	131.66	0.1950	0.2725	0.0000	132.13	125.17	1.06
16 FEB 89	Composite	66.05	0.0706	0.0000	0.0065	66.12	51.16	1.29

[a] OC-Organic carbon: includes carbon from semivolatile organics.

[b] EC-Elemental carbon.

[c] Carbon in HCN, volatile HC(C_1-C_{10}) and inorganic carbonate account for less than 0.01 percent, when combined.

[d] Ratio of total carbon mass measured to carbon mass in fuel.

[e] Number following date is detonation number of multidetonation trial.

[f] NS - not sampled.

5.4.2.1. These results are in sharp contrast to those predicted by certain theoretical models or determined during experiments involving limited available oxygen from surrounding air. The results of the BB TNT detonation tests are included, with some of these literature values, in Table 5.4. Clearly, OD of even the most oxygen-deficient of the common military explosives, TNT, yields a much higher fraction of CO_2 and lower fractions of CO and other carbonaceous products than models or earlier small-scale laboratory experiments have predicted (most of these assume oxidant is available only from the molecule and not from ambient air entrainment).

Table 5.4 Comparison of Carbon-Containing TNT Detonation Product Levels as Predicted by Literature and Determined by Experiment (kg/ton).

SPECIES	OB/OD[a]	COOK[b]	HAID/SCHMIDT[c]	ORNELLAS[d]	ORNELLAS[e]
Carbon dioxide	1.20×10^{3}	399.00	220.00	220.00	11.2
Carbon monoxide	4.07	25.40	239.00	221.00	658.00
Hydrogen cyanide	0.00	27.00	7.85	2.21	2.70
Carbon (soot)	5.77	154.70	159.00	175.40	47.9
Methane	2.45×10^{-2}	43.6	6.11	6.40	0.58
Ethane	-[f]			0.55	
Nonmethane HC	5.72×10^{-2}				
Semivolatile OC[g]	1.93				

[a] Present BB TNT OB work (density ≈ 1.4 g/cm^3). Values are estimates derived from mean emission factor data.

[b] Computed data for TNT (density = 1.59 g/cm^3).

[c] Experimental data from TNT detonation (density = 1.59 g/cm^3) from Haid and Schmidt (1931).

[d] Experimental data from confined TNT (density = 1.54 g/cm^3) under vacuum from Ornellas (1982).

[e] Experimental data from unconfined TNT (density = 1.54 g/cm^3) under vacuum from Ornellas (1982).

[f] C_2H_6 included in NMHC value of 5.72×10^{-2} kg/ton.

[g] Organic carbon from thermooptical analysis of aerosol/particulate collected on quartz-fiber filters.

5.5. Carbon-Balance Method

5.5.1. Calculating EF's

The carbon balance method of calculating EFs depends on two assumptions: (1) the mass of carbon available in the TNT can be accounted for in the masses of the various carbon-containing product species, and (2) the proportional distribution among carbon-containing products within individual microregions of the cloud remains relatively constant, even though the actual values for individual concentrations may be considerably different within different macroregions of the cloud.

5.5.1.1. Based on these two assumptions, the total volume of the cloud becomes irrelevant in making EF calculations, and the EF of any individual product *i* can be estimated by the equation:

Equation 5.1 Carbon Balance Emission Factor Determination.

$$EF_i = (f_c)\,\frac{[D_{ij}]}{[C_j]}$$

where,
f_c = mass fraction of carbon in the fuel (0.3701 for TNT),
$[D_{ij}]$ = average concentration of product *i* over any specific volume element *j* of the cloud, and
$[C_j]$ = concentration of all forms of carbon in the sample taken from volume element *j*.

5.5.1.2. For example, the maximum EF value calculated for 2,4-dinitrotoluene from analyses of 11 OD samples was 1.05×10^{-6} weight units (e.g., kg) of product per weight unit (e.g., kg) of TNT detonated. (Values used were $f_c = 0.3701$, $[D_{ij}] = 2.2 \times 10^{-4}$ mg/m^3, and $[C_j] = 77.9$ mg/m^3.)

5.5.2. Value in Field Testing

The carbon balance method has great potential for calculating OB/OD combustion product EFs in large-scale field tests, because total volumes of clouds and total concentrations of products over that whole "volume" do not need to be known; only "grab samples" need to be taken within the cloud by aircraft sampling, and CO_2 and target analyte levels above background need to be clearly measured.

5.6. Concentration Times Cloud Volume (C•V) Method.

5.6.1. Calculating EF's

The more traditional method of calculating an EF for product *i* is the C•V method.

Equation 5.2 Cloud Volume Emission Factor Determination

$$EF_i = \frac{[D_i]V}{M}$$

where: $[D_i]$ = average concentration of product *i* over the entire cloud immediately after detonation,
V = volume of the entire cloud, and
M = mass of TNT (or propellant) consumed.

5.6.2. Use of C•V

The use of this method is extremely difficult in the field environment because concentrations of products are not homogeneous over the entire cloud; therefore, many points in the diffusing cloud must somehow be sampled in order to obtain a true average product concentration. Also, it is difficult to accurately measure the actual volume of the diffusing, nonsymmetrical, ill-defined cloud at the precise time at which concentration samples are collected. These difficulties can be readily overcome in a chamber environment in which air concentrations and chamber volumes can be accurately determined as functions of sampling time. Thus, although difficult to apply in the field, this method was successfully used in calculating EFs from BB data. For example, the maximum EF calculated for 2,4-dinitrotoluene by this method was 0.819×10^{-6} weight units (e.g. kg) of the product produced per weight unit (e.g. 1 kg) of TNT detonated. (Values used were $[D_i] = 2.2 \times 10^{-4}$ mg/m^3, V = 833.8 m^3, and M = 0.224 kg or 224 g.)

5.6.2.1. Thus, the calculated carbon balance method EF (1.05×10^{-6}) for this compound compares extremely well with the C•V method EF of 0.819×10^{-6}.

5.6.2.2. As noted earlier, conducting these tests in an enclosed chamber environment allowed comparison of a proposed method of calculating these EFs (the carbon balance method) with the more traditional method involving multiplication of air emission concentration by the cloud volume (the C • V method). Results showed clearly that the proposed-carbon balance method, which is not dependent on cloud volume and therefore can be applied to cases in which the cloud volume is extremely difficult to estimate (such as after outdoor OD), gave EFs which were not statistically different from those obtained by the C • V method. This is certainly true when the cloud is homogeneous and its volume is accurately known (as in the BB). Even when the emission concentrations are nonhomogeneous (a condition which exists in outdoor OB/OD operations) in the chamber EFs from the carbon balance method were not different from the EFs obtained during the homogeneous period of sampling. The C • V method did not provide a good estimate of EFs when the emission concentrations are nonhomogeneous in the chamber. As a result, it was concluded that the carbon-balance method should be further evaluated in large-scale outdoor OB/OD operations. This assessment will determine the degree to which the principal carbon-containing species (CO_2) are above ambient background levels when the OB/OD clouds are sampled by FWAC, and the degree to which dilute, measurable concentrations of the volatile and semivolatile combustion products can be detected by analysis of FWAC samplers mounted on FWAC. If above-background CO_2 concentrations and detectable emission concentrations can be measured by analysis of FWAC-mounted samplers, then EFs can be calculated more accurately than by using methods requiring careful cloud volume estimations.

5.7. Emission Factors.

5.7.1. Calculations

After sample analysis concentrations, BB volumes, BB ventilation rates, fuel carbon contents, and total product carbon contents became known, EFs for each combustion product were calculated. These EFs are expressed as weight (e.g., kg) of each combustion product produced per unit weight (e.g., 1 kg) of explosive or propellent consumed. The maximum EFs detected are summarized in Tables 5.5a through 5.5d.

5.7.2. Carbon Dioxide

Tables 5.3 and 5.4 indicate that CO_2 was by far the dominant product in both TNT detonations and double-base and composite propellant burns.

5.7.3. Carbon Monoxide and Other Organic Compounds

The proportion of carbon monoxide, volatile organic hydrocarbons (C_1-C_{10}), total organic carbon, and elemental carbon decreases when the two types of propellants are burned. Emission factors for the exotic semivolatile organics rarely reached levels of 10^{-4} kg of emissions per kilogram of fuel; most are generated in quantities approximating 10^{-6} to 10^{-9} kg per kg of fuel. For this latter range of values, initial source strengths of emitted compounds would be in the range of 0.05 to 50 grams (10^{-3} to 10^{-6} lb) for a 907-kg (2000-lb) OB/OD operation. These source strengths, once dispersed by atmospheric meteorological conditions, even at short downwind distances, are not expected to create measurable quantities of the analytes at downwind receptor populations/locations.

5.7.4. Metals and Nonmetals

After TNT detonations or propellant burns, metals and nonmetals were rarely detected in concentrations high enough for EFs to exceed 10^{-4}, except in cases where the element was traceable directly to the fuel mixture (Al, Cu), igniter, chamber pan, or floor material, or previous, unrelated SNL test activities conducted in the BB.

Table 5.5a Maximum Emission Factors (kg/kg) and ppmw for Species Found for TNT Detonations, Double-Base, and Composite Propellant Burns.

Species	TNT Detonation		Double-Base Burn		Composite Burn	
	EF (kg/kg)	**ppmw**	**EF**	**ppmw**	**EF**	**ppmw**
Carbon dioxide	1.33	$1.33 \times 10^{+6}$	9.7×10^{-1}	$9.7 \times 10^{+5}$	4.18×10^{-1}	4.18
Carbon monoxide	7.17×10^{-3}	7170	9.54×10^{-4}	954	4.28×10^{-4}	4.28
Nitrogen dioxide	2.60×10^{-3}	2600	2.81×10^{-3}	2810	1.57×10^{-3}	1570
Nitric oxide	1.46×10^{-2}	14600	[illegible]47 x 10^{-2}	24700	5.37×10^{-3}	5370
Sulfur dioxide	2.23×10^{-4}	223	[illegible] x 10^{-4}	360	1.44×10^{-4}	144
Ozone[a]						
Methane	1.31×10^{-4}	131	7.26×10^{-5}	72.6	7.99×10^{-5}	79.9
Acetylene	1.82×10^{-5}	18.2	2.18×10^{-5}	21.8	5.27×10^{-5}	52.7
Benzene	8.67×10^{-6}	8.67	1.27×10^{-5}	12.7	3.02×10^{-5}	30.2
Paraffins[b]	1.45×10^{-4}	145	6.98×10^{-5}	69.8	6.95×10^{-5}	69.5
Olefins[b]	3.03×10^{-5}	30.3	7.94×10^{-5}	79.4	$3.93 \times 10_{-5}$	39.3
Non-benzene aromatic	2.99×10^{-5}	29.9	9.8×10^{-5}	98.5	1.98×10^{-5}	19.8
SEMIVOLATILE ORGANICS						
2,6-Dinitrotoluene	4.39×10^{-7}	0.439	1.43×10^{-8}	0.0143	3.72×10^{-9}	0.00372
4-Nitrophenol	2.59×10^{-6}	2.59	6.87×10^{-7}	0.687	4.08×10^{-7}	0.408
2,4-Dinitrotoluene	1.05×10^{-6}	1.05	$< 1.75 \times 10^{-10}$	<0.000175	1.04×10^{-8}	0.0104
2-Nitronaphthalene	6.43×10^{-7}	0.643	5.43×10^{-8}	0.0543	2.03×10^{-8}	0.0203
2,4,6-Trinitrotoluene	3.38×10^{-6}	3.38	5.01×10^{-5}	0.0501	$< 1.37 \times 10^{-9}$	<0.00137
1-Nitropyrene	1.06×10^{-6}	1.06	$< 1.66 \times 10^{-8}$	<0.0166	$1.98 \times 10_{-8}$	0.0198
1,6-Dinitropyrene	ND[c]	ND	ND	ND	ND	ND
Phenol	2.52×10^{-5}	25.2	4.39×10^{-6}	4.39	3.78×10^{-6}	3.78
Dibenzofuran	1.32	1.32	2.25×10^{-7}	0.225	2.79×10^{-7}	0.279

See Table 5.5d for notes

Table 5.5b Maximum Emission Factors (kg/kg) and ppmw for Species Found for TNT Detonation, Double-Base, and Composite Propellant Burns.

Species	TNT Detonation		Double-Base Burn		Composite Burn	
	EF (kg/kg)	ppmw	EF	ppmw	EF	ppmw
SEMIVOLATILE ORGANICS (cont'd)						
2-Naphthylamine	ND	ND	ND	ND	ND	ND
Diphenylamine	ND	ND	ND	ND	ND	ND
Benz[c]acridine	ND	ND	ND	ND	ND	ND
Benz[a]anthracene	$<3.30 \times 10^{-8}$	<0.033	$<1.83 \times 10^{-9}$	<0.00183	$<1.66 \times 10^{-8}$	<0.0166
Benzo[a]pyrene	3.01×10^{-6}	3.01	8.96×10^{-7}	0.896	5.23×10^{-8}	<0.0523
Dibenz[a,h]anthracene	1.73×10^{-6}	1.73	ND	ND	ND	ND
N-Nitrosodiphenylamine	1.23×10^{-6}	1.23	1.45×10^{-6}	1.45	3.45×10^{-8}	0.0345
Naphthalene	1.50×10^{-4}	150	$<1.55 \times 10^{-9}$	<0.00155	1.44×10^{-6}	1.44
1-& 2-Methylnaphthalene	3.00×10^{-5}	30				
1,3,5-Trinitrobenzene	2.75×10^{-9}	0.00275				
Biphenyl	$<5.20 \times 10^{-8}$	<0.052				
Phenanthrene	1.85×10^{-7}	0.185				
2,5-Diphenyloxazole	7.23×10	72.3				
1,1,3-Trimethyl-3-phenylindane	5.70×10^{-7}	0.57				
Pyrene	2.02×10^{-7}	0.202				
4-Nitrosodiphenylamine	---^{4}		ND	ND		
2-Nitrodiphenylamine			ND	ND		
4-Nitrodiphenylamine			ND	ND		
Nitroglycerin			ND	ND		
Resorcinol			ND	ND		
Di-n-propyl adipate			ND	ND		

See Table 5.5d for notes

Table 5.5c Maximum Emission Factors (kg/kg) and ppmw Found for TNT Detonation, Double-Base, and Composite Propellant Burns.

Species	TNT Detonation		Double-Base Burn		Composite Burn	
	EF (kg/kg)	ppmw	EF	ppmw	EF	ppmw
SEMIVOLATILE ORGANICS (cont'd)						
Triacetin			ND	ND		
Salicylic acid			ND	ND		
2,2-Methylene bis(4-methyl)-6-t-butylphenol					ND	ND
Phenyl di-isodecyl phosphite					ND	ND
5-ethyl-1,3-diglycidyl-5-methyl hydantoin diepoxide					ND	ND
Diethylenetriamine					ND	ND
Dioctyl sebacate					ND	ND
Isophorone di-isocyanate					ND	ND
METALS						
Aluminium					1.33×10^{-4}	133
Chromium	3.52×10^{-6}	3.52	BD	BD	4.77×10^{-6}	4.77
Iron					6.04×10^{-4}	604
Nickel	2.54×10^{-6}	2.54	BD	BD	1.59×10^{-6}	1.59
Copper			3.71×10^{-3}	3710		
Arsenic	BD	BD	BD	BD	BD	BD
Lead	1.97×10^{-5}	19.7	1.27×10^{-2}	12700	9.39×10^{-5}	93.9
Cadmium	2.86×10^{-6}	2.86	BD	BD	BD	BD
Antimony	1.06×10^{-6}	1.06	BD	BD	4.77×10^{-6}	4.77
Barium	9.31×10^{-4}	931	BD	BD	1.59×10^{-7}	159

See Table 5.5d for notes

Table 5.5d Maximum Emission Factors (kg/kg) and ppmw for Species Found for TNT Detonation, Double-Base, and Composite Propellant Burns.

Species	TNT Detonation		Double-Base Burn		Composite Burn	
	EF (kg/kg)	ppmw	EF	ppmw	EF	ppmw
BUBBLERS						
Ammonia	2.92×10^{-4}	292	BD	BD	BD	BD
Hydrogen cyanide	BD	BD	BD	BD	BD	BD
Hydrogen chloride					9.43×10^{-2}	94300

[a] Ambient ozone was reduced to near zero at detonation by reacting with the detonation products.

[b] For a full listing of compounds included within these groupings as analyzed by Oregon Graduate Center, refer to Table 3.4.4, this report.

[c] ND - detection limit was not determined.

[d] Blank Cell - not a target analyte during the cited detonation or burn.

[e] BD - target analyte not found in concentrations above detection limits. Detection limits (approximately 0.5 $\mu g/m^3$ for the elements corresponds to an EF of 2×10^{-6} for TNT and 1×10^{-6} for double-base and composite propellant). Detection limits (approximately 10 $\mu g/m^3$) for ammonia and hydrogen cyanide corresponds to an EF of 4×10^{-5} for TNT and 2×10^{-5} for double-base and composite propellant.

5.7.5. HCN, NH_3, and HCl

HCN was never detected during analysis of bubblers. NH_3 concentrations were detected slightly above background levels in only two TNT OD samples. These detections were also characterized by abnormally low values in the applicable background samples and are therefore speculative. As expected, HCl was measured in significant concentrations during air sampling from the composite fuel burn, however because of significant problems encountered with the HCL bubbler during sampling the data is considered unreliable.

5.7.6. Dioxins/Furans

A team from BCD specializing in sampling for polychlorinated dibenzodioxins (PCDD) and polychlorinated dibenzofurans (PCDF) sampled the chamber air following the burn of the composite propellant (the only propellant, explosive, and pyrotechnic (PEP) material containing chlorine tested in the BB), (Volume 2, Appendix B). The tetrachloro derivatives, particularly the symmetrically substituted 2,3,7,8-tetrachlorodibenzofuran and 2,3,7,8-tetrachlorodibenzodioxin isomers are of particular concern as toxicants. None of the most toxic 2,3,7,8-tetrachlorodibenzodioxin was detected and only one of two samples showed a barely marginal value for the less toxic 2,3,7,8-tetrachlorodibenzofuran showing that even its presence is problematical. Small amounts of some of the other, much less toxic, chlorinated derivatives were detected. The hepta-, and octa-CDD were detected in concentrations of 2.6 pg/m^3 and 15 pg/m^3 respectively. These concentrations result in EFs of 5.3×10^{-12}, and 30×10^{-12}. Using these EFs a similar mix of 10 metric tons of an AP composite propellant, when burned, would yield 53 μg of hepta-CDD, and 300 μg of octa-CDD.

5.7.7. Foam-Attenuated TNT Test

As might be expected, the foam-attenuated TNT detonation conducted 13 February 1989 produced generally lower concentrations of CO_2 and greater concentrations of CO, NO_2, NO, and volatile organics than did the various non-attenuated TNT detonations. The main effects of the foam were mitigation of the blast and noise, reduction of the total carbon mass recovery accounted for as CO_2, and the spreading of viscous, sulfonate-glycol/ether-xanthan polymer residue widely about the chamber floor.

SECTION 6. SAMPLE DISPERSION MODEL CALCULATION

6.1. The Volume Source Diffusion Model

The volume source diffusion model (VSDM) was used by the DPG Meteorology Division to estimate concentrations of emissions downwind from the detonation site. The dispersion model was applied to nominal source strengths of the combustion products as calculated from the maximum EF's determined from BB testing. Typical meteorological input parameters were chosen, based on conditions that provide a clearing index of at least 500 (a State of Utah test conduct requirement at DPG). The cloud height at stabilization for 907-kg (2000-lb) TNT detonations was calculated from the instantaneous cloud rise equations developed by National Aeronautic and Space Administration (NASA). The stabilization height is the calculated point relative to detonation at which the energy input from the detonation has been expended and the cloud growth and movement are thereafter only influenced by the ambient environment meteorological conditions.

6.2. Sample Calculations

The following sample calculations use the maximum EF for 2,4-dinitrotoluene, a TNT OD combustion product of interest. The maximum EF calculated for this compound, based on BB results, was 1.05×10^{-6}. Multiplying the EF by the weight of TNT to be detonated yields the maximum total source strength of that compound. Thus, the amount of 2,4-dinitrotoluene resulting from a 907-kg (2000-lb) TNT detonation would be approximately 1 gram (1×10^{-3} kg). Figure 6.1 shows that the maximum momentary peak ground-level 2,4-dinitrotoluene concentration (1.1×10^{-3} $\mu g/m^3$) would be detected approximately 2.5 km downwind from the detonation site and should not have persisted for more than a few minutes. The maximum ambient air concentration limits for 2,4-dinitrotoluene exposure to the general population have been set by several states. One of the most restrictive is presently 15 $\mu g/m^3$, set by North Dakota for an 8-hour exposure. Thus, for the 2,4-dinitrotoluene expected from a 907-kg (2000-lb) TNT open detonation, there would be essentially no risk to the receptor site/general population since the peak concentration at ground level (at approximately 2.5 km downwind) would be 14,000 times less than the North Dakota ambient air standard.

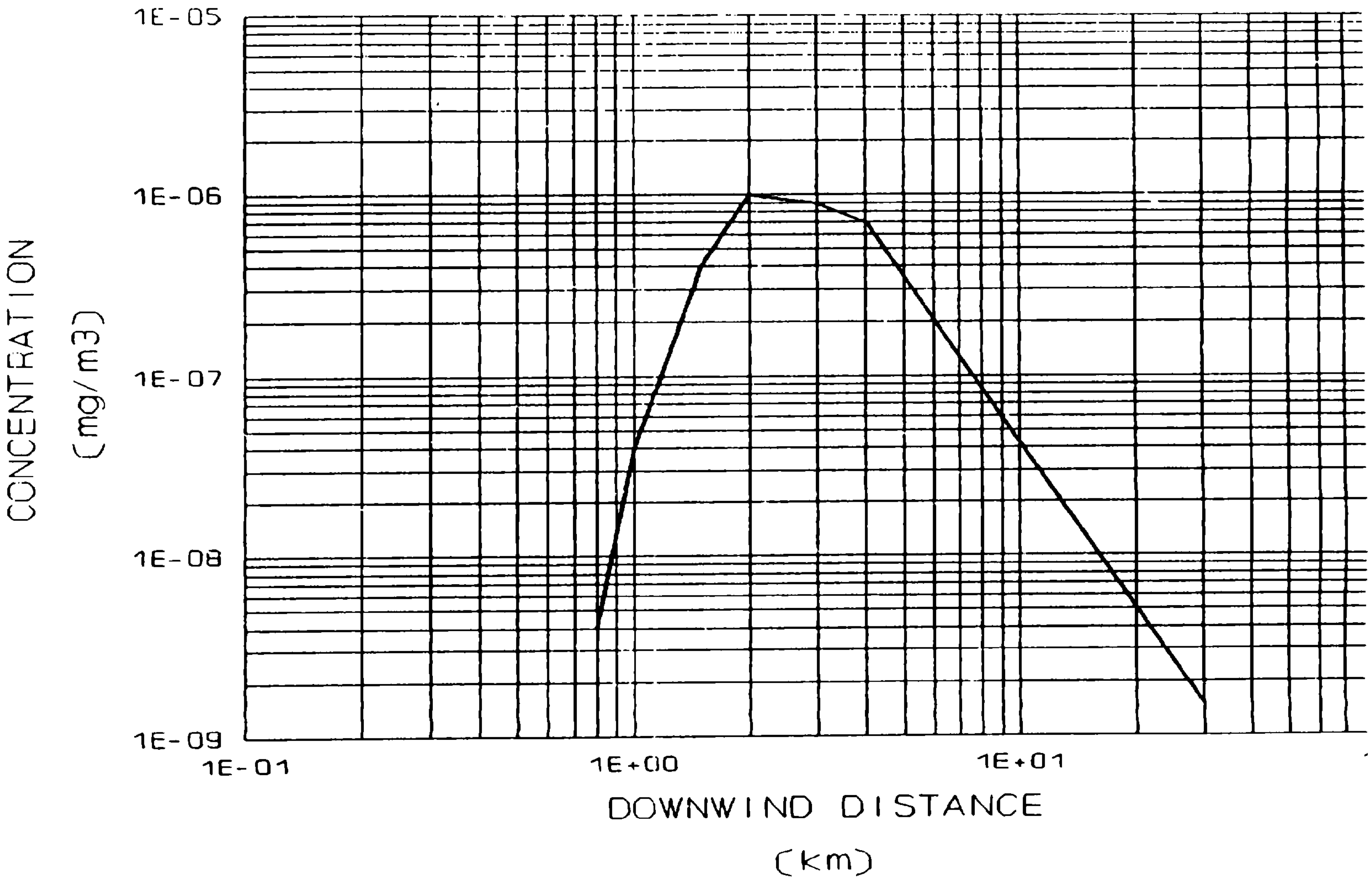

Figure 6.1 Calculated Profile of Peak Concentration at Ground Level Versus Downwind Distance for 2,4-DNT.

6.2.1. Calculation Method

6.2.1.1. Criterion/Restriction: 15 $\mu g/m^3$ ambient air concentration limit for an 8-h time period for the general population, North Dakota.

6.2.1.2. Maximum EF for 2,4-dinitrotoluene: 1.05×10^{-6} kg/kg for TNT detonation.

6.2.1.3. Maximum peak 2,4-dinitrotoluene concentration calculated from application of the VSDM for a 1×10^{-3} kg source: approximately 1.1×10^{-6} mg/m^3 (1.1×10^{-3} $\mu g/m^3$).

Equation 6.1 Equation Used To Calculate Safety Factor for 2,4-DNT.

$$\frac{15\ \mu g/m^3\ \textit{(general population concentration limit, ND)}}{1.1\ x\ \mu g/m^3\ \textit{(momentary peak concentration max at ground level 2.5 km det.)}} = 1.4x10^4\ \textit{(about 14,000} \times \textit{less than allowable ND standards)}$$

6.3. Risk Assessments

No attempt has been made in this report to prepare detailed, formal risk assessments based on the source strengths of each projected product derived from the BB data. However, there appears to be sufficient data here to support such analyses by applying concepts similar to those outlined in the November 1988 EPA Report, Risk Assessment Guidelines and Information Directory, the October 1989 EPA Draft Final Report, Background Information Document for the Development of Regulations for PIC Emissions from Hazardous Waste Incinerators, and other similar documents pertinent to implementation of ambient air quality standards.

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SECTION 7. CONCLUSIONS

7.1. Conclusions (Addressed by Study Objectives)

7.1.1. Objective 1 - BangBox Characterization: Characterize the BB chamber volume, ventilation rate, and combustion product cloud homogeneity level.

The chamber volume, ventilation rate, and cloud homogeneity were successfully determined, and found satisfactory for subsequent use throughout the data evaluation/analysis processes.

7.1.2. Objective 2 - Sampling and Analyses: Develop and improve proposed air sampling equipment and sample analysis procedures to be used in later phases on the FWAC, for sampling product clouds from large-scale follow-on outdoor OB/OD trials.

7.1.2.1. Samplers and detectors used in the BB tests that were felt to have performed well enough to be used for FWAC sampling of open-air clouds of OB/OD products included 0.85-L evacuated canisters; 6-L evacuated canisters; 32-L evacuated tanks; CO_2, CO, SO_2, and NO_x real-time gas monitors; quartz-fiber particulate filters; Teflon™ particulate filters; the Belfort integrating nephelometer; the RAM nephelometer; and DMPS, ASASP, and FSSP aerosol spectrometers.

7.1.2.2. Samplers used in BB tests that were felt to have performed marginally included the resin filters (both Porapak-R™ and XAD-2™) which were an integral part of the semi-VOST system and the Velostat™ bag. The resin filters greatly constricted airflow, thus limiting their suitability for FWAC grab sampling. The bag proved inadequate for some volatile and semivolatile organic compounds, because of absorption and subsequent off-gassing.

7.1.2.3. The CO real time instrument did not perform properly. The CO data was obtained from the 6-L canister analysis.

7.1.3. Objective 3 - Comparison of SFC/MS and GC/MS: Refine, standardize, and compare SFC and GC techniques for extracting and analyzing resins, and filters, for trace quantities of semivolatile organic OB/OD combustion products and residues, using MS detectors.

Both SFC/MS and GC/MS are sufficiently sensitive analysis techniques to detect and quantify semivolatile organic (exotic) target analytes collected from chamber air by quartz-fiber filters, resin-filled canisters, and evacuated stainless steel cylinders. SFC is superior for some of the less thermally stable exotic species of interest, e.g. N-nitrosoamines, and some of the nitro compounds.

7.1.4. Objective 4 - Other Standard Analytical Methods: Verify adequacy of other standard analytical methods to be used for analyses of gases, particulates, volatile organic compounds, metals, and nonmetals.

7.1.4.1. The Velostat™ bag sampler proved inadequate for some volatile and semivolatile organic compounds because of absorption and subsequent off-gassing problems.

7.1.4.2. The liquid impingers (bubblers) used for measuring HCN, NH_3, and HCL in the cloud were adequate for the BB.

7.1.4.3. The extraction and analytical procedures (GC/FID) used for analysis of the volatile organics (VOC's) and the SF_6 (GC/ECD) proved to be highly successful.

7.1.4.4. Elemental analysis proved successful using XRF techniques.

7.1.5. Objective 5 - Identify and Quantify Specific Target Analytes: Identify and quantify specific target analytes for TNT, a double-base propellant, and a composite propellant.

7.1.5.1. TNT Detonation

7.1.5.1.1 The maximum EF values calculated from TNT detonation data are given in Table 7.1.

Table 7.1 Maximum Emission Factors From TNT Detonation by the Carbon Balance Method.

Species	Emission Factor (kg/kg)
Methane	1.3×10^{-4}
Acetylene	1.8×10^{-5}
Benzene	8.7×10^{-6}
Selected C_2-C_{10} non-methane paraffins	1.5×10^{-4}
Selected C_2-C_{10} olefins	3.0×10^{-5}
Selected non-benzene aromatics	3.0×10^{-5}
Phenol	2.5×10^{-5}
Naphthalene	1.5×10^{-4}
Other individual semivolatile (target analyte exotic) aromatics varied from	3×10^{-9} to 7×10^{-5}

7.1.5.1.2 Open detonation is an extremely efficient TNT thermal treatment method. Carbon-containing species measured from 227-gram (0.5-lb) TNT detonations were generally distributed as shown in Table 7.2.

Table 7.2 Distribution from Carbon-containing Species Measured From TNT.

Species	Percent
Carbon Dioxide	97.20
Carbon Monoxide	0.50
C_1 to C_{10} volatile hydrocarbons and other organics	0.57
Elemental carbon (soot)	1.71

7.1.5.2. Propellant Burn

7.1.5.2.1 Propellant burn maximum EFs were generally one to two orders of magnitude lower than those for the corresponding TNT detonation product.

7.1.5.2.2 Based on propellant carbon conversion to CO_2 open burning of double-base and composite propellants is an extremely efficient thermal treatment method. However, further work is needed to determine the fate of chlorine in the combustion products. Carbon-containing species measured from burning these fuels are distributed as shown in Table 7.3.

Table 7.3 Carbon-containing Species Measured from Propellant Burns.

Species	**Percent**	
	Double-Base	**Composite**
Carbon Dioxide	99.64	99.88
Carbon Monoxide	0.15	0.11
Organic Carbon	0.21	0.00
Elemental carbon (soot)	0.00	0.01

7.1.6. Objective 6 - PCDD's and PCDF's: Assess polychlorinated dibenzodioxins (PCDDs) and dibenzofurans (PCDFs) levels generated from burning the composite propellant containing high concentration of NH_4ClO_4.

None of the most toxic PCDDs were detected, and only one of two samples had a marginal value for the less toxic PCDFs indicating that their source was not derived from the composite propellant.

7.1.7. Objective 7 - Morphology, Composition and Size Distribution of Particulate: Provide information on the morphology, composition, and size distributions of airborne particulate material generated by OB/OD operations in the BB.

Particulate morphology and composition was accomplished by SEM and optical microscopy. The results showed that over 90 percent of the particulate was soot (carbon) with small amounts of calcium carbonate and non-asbestos insulating material.

7.1.8. Objective 8 - Carbon Balance Method: Examine, using data produced under controlled conditions, the validity of the proposed carbon balance method of calculating emission factors; compare the results with those calculated using the more-conventional cloud volume times concentration method.

7.1.8.1. The proposed carbon balance method of calculating emission factors of products of combustion resulting from OD of TNT and double-base and composite propellant burns has been verified under conditions which permit a careful comparison with the cloud-volume method.

7.1.8.2. EFs calculated by the carbon balance-method agreed within experimental uncertainty with those calculated by the more traditional concentration times cloud volume method. During periods of nonhomogenity of cloud concentration the carbon balance-method provided better estimates of the EF.

7.1.8.3. Sufficient CO_2 concentrations (above background ambient levels) must be measurable to be able to apply the carbon balance method of calculating EFs during followon, large-scale outdoor OB/OD tests.

7.1.9. Objective 9 - QA/QC Procedures: Identify or develop appropriate program-specific QA/QC procedures.

A QA/QC program was developed specifically to address BB testing and subsequent analyses. This program, along with its findings and conclusions, is delineated in Volume 3 of this report.

7.1.10. Objective 10 - Sample Storage/Transport Procedures: Establish procedures for transport and storage of sample specimens.

Procedures were established to ensure that, during transportation and storage, semivolatile compounds would be retained by their respective sampling media, and that sample identity and integrity would be maintained. These procedures are described in Volume 3 of this report.

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SECTION 8. RECOMMENDATIONS

8.1. Particulate Counting and Sizing Measurements and Data Handling Procedures

Further review particulate counting and sizing measurements and data handling procedures for adequacy before conducting full-scale, field OB/OD operations.

8.2. Bag Sampling for the FWAC

Find a more suitable bag for FWAC use during later phases of the OB/OD program. Absorption and off-gassing properties of this replacement bag should be well-characterized. If this is not available, replace the bag with another collection media.

8.3. Semi-VOST Resin Media Sampling

Replace the semi-VOST resin media with another collection media.

8.4. SFC/MS Analytical Methods

Expand the SFC/MS method of analysis to embrace samples derived from soil.

8.5. HCL, HCN, and NH_3

Define and authenticate a means of adequately measuring HCL, HCN, NH_3 in field OB clouds from a FWAC platform before conducting large-scale composite propellant burns.

8.6. Carbon Balance Method

Conduct large scale field tests of OB/OD PEP material to obtain EFs based on the carbon balance technique proven in the BB test.

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APPENDIX A - CONSOLIDATED ABBREVIATIONS

ACGIH	American Conference of Governmental Industrial Hygienists
AEHA	U.S. Army Environmental Hygiene Agency, Aberdeen Proving Ground, Maryland
AFB	Air Force Base
AMC	U.S. Army Materiel Command, Alexandria, Virginia
AMCCOM	U.S. Army Armament, Munitions and Chemical Command, Rock Island, Illinois
amino-PAH	aminopolycyclic aromatic hydrocarbons
ANOVA	analysis of variance
AP	ammonium perchlorate
APS	aerodynamic particle sizer
ASASP	active scattering aerosol spectrometer probe
AWL	Alpine West Laboratories, Provo, Utah
BB	BangBox
BCD	Battelle Columbus Division, Columbus, Ohio
BD	target analyte not found in concentrations above detection limits
BYU	Brigham Young University, Provo, Utah
CAA	Clean Air Act
CDD	chlorinated dibenzodioxin
CDF	chlorinated dibenzofuran
CI-SIM	chemical ionization, selective-ion monitoring
CSI	Columbia Scientific Instruments
C•V	concentration times cloud volume method
CWA	Clean Water Act
DMC	Data Management Center
DMPS	differential mobility particle sizer
DoD	Department of Defense
DPG	U.S. Army Dugway Proving Ground, Dugway, Utah
EC	electron capture *or* elemental carbon
ECD	electron capture detector
EDAX	energy-dispersive X-ray analysis
EER	Energy and Environmental Research Corporation, Irvine, California

EF	emission factor(s)
EI	electron impact
EI-MS	mass spectrometer used in the electron impact ionization mode
EI/MS	electron impact ionization/ mass spectrometry
EIS	environmental impact statement
ELI	Environmental Labs, Incorporated, Provo, Utah
EOD	explosive ordnance disposal
EPA	U.S. Environmental Protection Agency
EPO	Environmental Protection Office, U.S. Army Dugway Proving Ground, Dugway, Utah
ER	expansion ratio
FID	flame ionization detector
FSSP	forward scattering spectrometer probe
FTIR	Fourier Transform Infrared Spectrometry
FWAC	fixed-wing aircraft
GC	gas chromatograph(y)
GC-ECD	gas chromatography with an electron capture detector
GC-FID	gas chromatography with a flame ionization detector
GC/MS	gas chromatography-mass spectrometry
GLP	good laboratory practices
HE	high explosive
HMX	octamethylenehexanitramine
HNBB	hexanitrobibenzyl
HRGC/HRMS	combined capillary column gas chromatography/high resolution mass spectrometry
HS	high-speed
LASD	Los Angeles Sheriff Department
LBL	Lawrence Berkeley Laboratory, Berkeley, California
LC	liquid chromatography
LOD	limit of detection
LOI	letter(s) of instruction
NO_x	nitrogen oxide (s)

MR	multiple range
MRI	Midwest Research Institute, Kansas City, Kansas
MS	mass spectrometry (or mass spectrometer)
MSA	Mine Safety and Appliance Company
NA	not targeted for analysis *or* not applicable
NASA	National Aeronautical and Space Administration
NATICH	National Air Toxics Information Clearinghouse
NBS-SRM	National Bureau of Standards (now NIST)- Standard Reference Material
ND	no data *or* detection limit not determined
NEPA	National Environmental Policy Act
NF	not found in the sample matrix *or* not determined
NIST	National Institute of Science and Technology
nitro-PAH	nitropolycyclic aromatic hydrocarbons
NIOSH	National Institute for Occupational Safety and Health
NOSIH	Naval Ordnance Station, Indian Head, Maryland
NO_x	nitrogen oxides
NS	not sampled
OB	open burning
OB/OD	open burning/open detonation
OC	organic carbon
OD	open detonation
OGC	Oregon Graduate Center, Beaverton, Oregon
OSHA	Occupational Safety and Health Administration
PAH	polycyclic aromatic hydrocarbons
PANH	polycyclic aromatic nitrogen heterocycles
PAOH	polycyclic aromatic oxygen heterocycles
PCDD	polychlorinated dibenzodioxins
PCDF	polychlorinated dibenzofurans
PETN	pentaerythritol tetranitrate
PEP	propellants, explosives, and pryotechnics
PIC	products of incomplete combustion
PICI/SIM	Positive ion chemical ionization/selective ion monitoring

PID	photoionization detector
PIP	product improvement program
PM	program manager
PMS	Particle Measuring Systems, Inc.
PUF	polyurethane foam
QA	quality assurance
QA/QC	quality assurance/quality control
QC	quality control
QAA	quality assurance agency
QAPP	quality assurance project plan
QAU	quality assurance unit
RCRA	Resource Conservation and Recovery Act
RDX	hexamethylenetrinitramine
RFD	Reno (Nevada) Fire Department
RIC	relative ion count
RSD	relative standard deviation
RTP	Research Triangle Park, North Carolina
SDPDA	Special Defense Property Disposal Account
semi-VOST	semivolatile organic sampling train
SEM	scanning electron microscope/microscopy
SFC	supercritical fluid chromatography
SFC/MS	supercritical fluid chromatography/mass spectrometry
SF_6	sulfur hexafluoride
SIM	selected-ion monitoring (or selective-ion monitoring)
SNL	Sandia National Laboratories, Albuquerque, New Mexico
SOP	standing operating procedures
SS	stainless steel
SSC	stainless steel canister
SSL	Sunset Laboratory, Forest Grove, Oregon
STEL	short-term exposure limit
STP	standard temperature and pressure (25°C and 760 torr)
TCD	thermal conductivity detector

TDP	test design plan
TEAD	U.S. Army Tooele Army Depot, Tooele, Utah
TECO	Thermo Electron Instruments (Company)
TECOM	U.S. Army Test and Evaluation Command, Aberdeen Proving Ground, Maryland
THC	total hydrocarbon
TLV	threshold limit values
TNT	2,4,6-trinitrotoluene
TSC	technical steering committee
TSP	total suspended particulate
TWA	time-weighted average
USATHAMA	U.S. Army Toxic and Hazardous Materials Agency, Aberdeen Proving Ground, Maryland
UV	ultraviolet
VOC	volatile organic compounds
VSDM	Volume Source Diffusion Model
XRF	X-ray fluorescence *or* X-ray fluorescence spectrometer

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