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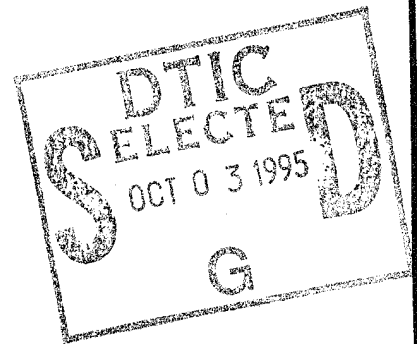


LAUNCH VEHICLE ABORT SOURCE STRENGTH MODEL-
PROPELLANT INTERACTION STUDY

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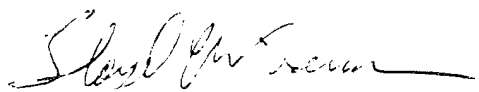
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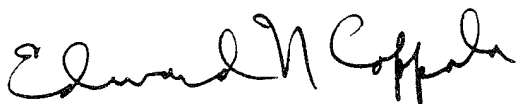
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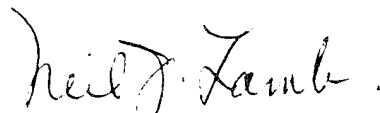
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Launch Vehicle Abort Source Strength Model - Propellant Interaction Study

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DISTRIBUTION AVAILABILITY STATEMENT

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A series of tests involving the combustion of solid and liquid rocket propellants used to fuel the Titan II, Titan IV and Delta II launch vehicles was performed. The purpose of these tests was to obtain chemical combustion data to support modeling of launch vehicle accidents. Propellants tested were Aerozine-50, dinitrogen tetroxide, RP-1, liquid oxygen, PBAN, and Castor IVA. Tests were conducted in a 150-liter stainless steel combustion chamber in air at nominal pressure (0.8 atmospheres at Denver barometric conditions). Measurements of the chamber gas temperature and internal pressure were taken and gas samples were withdrawn and analyzed for combustion gases, unreacted propellants, unreacted oxygen and organic vapors. A witness plate was used to collect condensed residues. Results from these tests suggest different chemical fates of these propellants than those predicted by theory alone.

Propellant; Combustion; Reactions; Aerozine-50;
Dinitrogen tetroxide; RP-1; PBAN; Castor IVA;
Oxygen; Condensates

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PREFACE

This final report was prepared by Martin Marietta Astronautics, PO Box 179, Denver, Colorado 80201 in accordance with CDRL Data Item Number A004 of Attachment 1 to Contract F08635-92-C-0056 (Martin Marietta Proposal Number P91-60091-3) as authorized by Contract Award dated 92 Aug 10 for the Armstrong Laboratory, Tyndall Air Force Base, Florida 32403. Efforts documented in this report were performed between October 1992 and May 1993. Captain Floyd Wiseman was the AL/EQS-OL project officer.

The author would like to acknowledge the Martin Marietta test engineering, test operations, and instrumentation support groups at the Engineering Development Laboratory for their assistance in the conduction of these tests, and Martin Marietta personnel at the Chemical Technology Laboratory for the identification and measurement of chemical reaction products.

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EXECUTIVE SUMMARY

A. OBJECTIVE

The objective of this study was to evaluate gases and condensates resulting from mixing and reacting various amounts of liquid and solid rocket propellants, and to apply the derived data to predicting emissions from launch vehicle accidents. The use of this data to modify existing source models developed by theoretical methods was also of interest to this study.

B. BACKGROUND

Launch vehicle accidents, such as the Titan 34 D-9 explosion at VAFB in April 1986, result in the formation of hot and thermally buoyant fireballs, release considerable amounts of thermal energy, and emit a variety of chemicals into the atmosphere. These chemicals consist primarily of combustion products from the reacted liquid and solid rocket propellants, propellant decomposition products, and vaporized propellants. Release of these materials into the atmosphere can produce airborne concentrations of toxic chemicals which exceed local and federal environmental health regulations.

Calculations of toxic hazard corridors (THCs) for launch vehicle accidents are required to support pre-launch risk assessment, in-flight disaster response support, and, in the case of a catastrophic accident, accident response and damage assessment. Studies conducted previously, such as Project Pyro tests completed in 1968, were useful in describing vehicle failure modes, fireball sizes, and heat releases from these types of accidents. Data from these tests, however, showed a high degree of variability and did not address the nature or amounts of released chemicals. Uncertainties in cloud composition and temperature have forced conservative estimates of THCs, which in turn have restricted launch operations. More reliable and defensible THC estimates are required to support launch operations and to protect public health.

C. SCOPE

This document presents the methodology and results of laboratory tests used to identify chemical reaction products resulting from liquid and solid rocket propellants. Section I provides an introduction to the technology. Section II discusses the approach taken in the laboratory tests, a brief description of propellant chemistry of interest to this study, a description of the test design and associated hardware, and the chemical analysis methods employed. Section III discusses the test results. This section includes results from pre-test measurements of the combustion chamber, results from chemical recovery tests, and results from the propellant combustion tests. Section IV presents a discussion of the results. Conclusions and recommendations are presented in Sections V and VI, respectively. Supporting documentation, such as test data sheets, a schematic of the combustion chamber, calculations for sizing the nozzles, and a description of the chemical analysis plan are presented in the Appendixes.

D. METHODOLOGY

To simulate chemical reactions from launch vehicle accidents, test methods were selected to approximate the environmental conditions expected in an in-flight accident. These propellant reactions occur in air at ambient conditions of temperature, pressure, and relative humidity. For these reasons, a stainless steel chamber was constructed and sized such that the propellants could be combusted in an air atmosphere, and that after combustion, the reaction products could cool by natural means to ambient temperature. The chamber was also sized such that the resulting pressure, after cooling of combustion gases, was approximately one atmosphere total pressure.

Chemical analysis methods were selected, or developed as needed, for chemicals of special interest to this study. These chemicals include hydrazine, unsymmetrical dimethylhydrazine, nitrogen dioxide, ammonia, hydrogen, oxygen, nitrogen, methane, hydrogen chloride, carbon dioxide and carbon monoxide. These chemicals occur from propellant combustion reactions, from the thermal decomposition of excess liquid propellants, and from the vaporization of excess liquid propellants.

E. TEST DESCRIPTION

Propellants tested in this study include Aerozine-50 and dinitrogen tetroxide (liquid propellants used to fuel the Titan II and Titan IV launch vehicles), PBAN solid rocket propellant used on the Titan IV Solid Rocket Motor, RP-1 and oxygen (liquid propellants used to fuel the Delta II launch vehicle), and Castor IVA solid rocket propellant used on the Delta II first stage thrust augmentation. These propellants were reacted in a 150-liter stainless steel combustion chamber in air at nominal pressure (0.8 atmospheres at Denver barometric conditions). The hypergolic liquid propellants (Aerozine-50 and dinitrogen tetroxide) were injected into the chamber under pressure through square-edged orifices and impinged directly on each other, to initiate and sustain combustion. The Delta II propellants, RP-1 and gaseous oxygen (GOX), likewise were injected into the chamber under pressure and intersected at a high energy spark-gap which was used to ignite these propellants. Liquid propellants were tested under fuel-rich, oxidant-rich, and stoichiometric conditions. Solid propellants were ignited by passing a current through a high resistivity nichrome wire imbedded in the propellant.

Measurements of the internal gas temperature, chamber wall temperature, and internal pressure were taken during the course of the combustion process. In addition, gas samples were withdrawn from the chamber and analyzed using standard chemical methods for expected combustion gases (carbon dioxide, carbon monoxide, hydrogen, hydrogen chloride, and nitric oxide), unreacted propellants (hydrazine, unsymmetrical dimethylhydrazine, nitrogen dioxide, and hydrocarbons), organic chemicals, and oxygen reacted from the air. A stainless steel witness plate was also placed inside the chamber to collect and analyze condensates which formed during the course of the propellant combustion tests. The identification of toxic chemicals, both in the gas and condensed phases, was of particular interest in this study.

F. RESULTS

Results from this study suggest significantly different chemical fates for some of the liquid rocket propellants than those predicted by chemical theory only. Aerozine-50 and carbon monoxide experienced significant afterburning with available oxygen. Carbon dioxide was predominately formed in the latter case. Residual unreacted nitrogen dioxide was present in all tests involving dinitrogen tetroxide, even the stoichiometric condition. Residual hydrazine (N_2H_4) and unsymmetrical dimethylhydrazine (UDMH) were absent in the vapor phase after completion of all the tests, including tests employing an excess of Aerozine-50. In addition, excess UDMH was not oxidized to carbon dioxide (CO_2) as expected. Incomplete reaction of the hydrazines and the formation of complex condensates had occurred. Thermal decomposition products of Aerozine-50 (ammonia, hydrogen, and methane) were also absent, suggesting that if these compounds formed, they had also reacted with air. Nitrosodimethylamine (NDMA), an oxidation product of UDMH and established animal carcinogen, was observed in many of the Aerozine-50/dinitrogen tetroxide tests. When these liquid propellants were combusted with the Titan IV solid propellant (PBAN), a substantial quantity of bronze-colored residue formed, and this residue produced spectral characteristics similar to ammonium nitrate. The combustion of RP-1 kerosene fuel with gaseous oxygen produced varying results, ranging from near complete combustion (for fuel-rich conditions), to negligible combustion (for oxidant-rich conditions). The oxygen from the air contributed to continued burning of the RP-1 in the fuel-rich test. When combined with the Delta II solid propellant (Castor IVA), combustion efficiency of all propellants declined, again suggesting the formation of complex condensates, including some chlorinated hydrocarbons.

G. CONCLUSIONS

These test results suggest that several chemical events are likely to occur in a launch vehicle accident. First, due to the difficulty in efficiently combusting Aerozine-50 with dinitrogen tetroxide, excess dinitrogen tetroxide is expected to be released into the atmosphere. This propellant is likely to be present as the nitrogen dioxide monomer due to thermodynamic considerations, and is especially important because of its toxicity. Unfortunately, the fate of released Aerozine-50, consisting of an equal weight mixture of hydrazine and unsymmetrical dimethylhydrazine, is not clear. Incomplete combustion of these fuels is also expected in a launch vehicle accident, however vaporization and thermal decomposition of the excess propellants were not substantiated in this test program. Catalytic decomposition on the chamber walls and the formation of complex condensates including hydrazones and nitrosamines were suspected. Secondly, the appearance of condensates between Aerozine-50 and solid rocket propellants is expected to be significant. Test results indicate the formation of a compound similar to ammonium nitrate may be expected. Finally, many of the residual fuel sources, such as Aerozine-50 and RP-1 are expected to undergo significant afterburning with air before dispersion processes can begin.

H. RECOMMENDATIONS

Further studies are recommended to evaluate the nature of condensed residues in further detail and to examine the fate of unreacted Aerozine-50 fuel, for which a mass balance could not be established under this present effort. Results of these studies would help elucidate the nature and extent of chemical reaction products not established in the present study, improve predictive methods, and enhance existing atmospheric dispersion models.

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SECTION I

INTRODUCTION

A. OBJECTIVE

Military spacecraft launches at the Eastern and Western Test Ranges require pre-launch risk analyses, in-flight disaster response support, and, in the case of a catastrophic accident, abort damage assessment. Calculation of toxic hazard corridors (THCs) is an important element of each of these requirements. Reductions in allowable public exposure to toxic vapors released in launch accidents threaten to impose constraints on launch operations as THCs may reach civilian populations more frequently than in the past.

Current atmospheric dispersion models used for establishing launch support THCs depend on accurate estimates of toxic vapor source strength to produce accurate "footprints" of downwind vapor concentration. Uncertainties in source cloud composition for various accident scenarios, especially those involving fire, explosion, and vehicle destruction, force conservative assumptions that may extend THCs and unnecessarily restrict launch opportunities. This makes development of accurate, defensible estimates of source cloud characteristics imperative for launch safety planning and emergency response.

Previous efforts related to source modeling for liquid- and solid-fueled rockets were based on an engineering analysis of the propellant systems, using Gibbs Free Energy (GFE) minimization to determine equilibrium chemical compositions. In addition, because not all chemical species, such as unreacted liquid propellants, could be accounted for using this theoretical approach, estimates on the amounts of these unreacted materials were necessary. These tests were undertaken to refine and amend the developed source model using derived empirical data. The chemical fate of unreacted liquid propellants, especially Aerozine-50 and dinitrogen tetroxide, are especially important in source term modeling and environmental risk assessment. Identification of other toxic chemicals, especially those not predicted by theoretical analyses, also assist in the accurate determination of toxic chemicals released from a launch vehicle accident.

The objectives of the propellant interaction tests are:

1. To study the expected chemical release from an accident involving a Titan II, Titan IV, or Delta II launch vehicle in which combustion products from the different types of propellants are mixed and reacted with each other and the surrounding air;
2. To use data from the laboratory tests to verify selected reaction products predicted by thermochemical analyses, with special emphasis on those products known to be toxic to humans.
3. To obtain other relevant test data, such as temperature and pressure changes after combustion, and relate these to predicted values; and
4. To use laboratory data to modify the thermochemical source model as required.

B. BACKGROUND

In 1982, the Air Force, along with other agencies, investigated expected toxic chemical source strengths and downwind compositions arising from an accident involving the Titan II operational weapons system. That study investigated chemical interactions and meteorologically induced atmospheric dispersion processes during an accident in which liquid dinitrogen tetroxide mixed with liquid Aerozine-50, resulting in a hypergolic explosion. Data obtained during the study included fireball temperatures, heat fluxes, diameters, gas product compositions, gas densities, and tabulations of thermochemical properties for hypergolic propellants and reaction products.

In 1983, a comprehensive study was funded by the Air Force to obtain, evaluate, and compile all pertinent existing data on hazards to Department of Defense/National Aeronautics and Space Administration (DOD/NASA) satellite and operational launch vehicles from liquid and solid propulsion systems. Results of this study were incorporated into a systems users' manual entitled Space Propulsion Hazards Analysis Manual (SPHAM), and included documentation on numerous vehicle types and propellant combinations.

As a result of an in-flight explosion of a Titan 34D from the Vandenberg launch complex in April 1986, another study was funded by the Air Force to determine toxic chemical emissions from this vehicle. This study was unique, in that the contribution of combustion products of the solid rocket booster components to the thermal and chemical environment of the resultant fireball were determined.

On August 10, 1992, Martin Marietta was awarded an Air Force contract to model combustion clouds resulting from an accident or range safety destruction involving a Titan II, Titan IV, or Delta II launch vehicle. As part of this contract, a more detailed investigation of credible failure modes and propellant mixing phenomena for these launch vehicles was required. As part of this study, laboratory scale tests were performed to validate the source model and to investigate the formation of chemicals previously unreported in the literature.

C. SCOPE

1. Required Data

The primary data required from the laboratory test program include the identification and analysis of target chemicals. A maximum of seven chemicals per test are authorized as part of this contract. In addition, identification of other chemicals, which may be present in the gas phase or condensed phase, are required. These chemicals may form in cross-reactions between solid and liquid propellants, and those chemicals which may be a human health concern are of special interest in this investigation.

Fourier Transform Infrared (FTIR) Spectroscopy and Gas Chromatography/Mass Spectrometry (GC/MS) were selected as preferred techniques for the identification of these chemicals. Chamber gas temperature and pressure measurements were taken during the course of the combustion reaction to evaluate thermal and pressure environments of the reacting propellants, and to compare these data with predicted values. Finally, visual observation of the combustion process was afforded by the use of a sight glass installed in the chamber. This enabled the test conductor to verify that the desired propellant ignition occurred and that the combustion process continued to completion.

2. Accident Conditions

The ability to evaluate the combustion characteristics of mixtures of solid and liquid propellants and to incorporate these data into a thermochemical model for a launch vehicle accident is highly dependent on the test design. Such parameters as reaction chamber geometry, heating and cooling effects, mixing of combustion gases, environmental conditions existing at the time combustion is initiated (pressure, temperature, relative humidity), propellant feed rates, and fuel/oxidant mixture ratios all influence the final product composition. The design goal of the propellant tests, therefore, is to model the behavior of these released propellants in such a manner that the results are representative of true accident conditions. In addition, the test design should facilitate scaling of the laboratory data to launch vehicle accidents.

The combustion clouds resulting from a launch vehicle accident depend on the failure modes (confined by missile, confined by ground surface, command destruct, low velocity impact). For test purposes, however, the following accident conditions for all launch vehicle failure modes were used as the baseline for the test design.

a. For the liquid hypergols, Aerozine-50 (A-50) and dinitrogen tetroxide (N_2O_4), only a fraction of the liquids are expected to participate in the combustion reaction. The remainder are vaporized and contribute to the toxic composition of the combustion cloud. Previous studies have placed the fraction of hypergols involved in the combustion reaction at 10-20 percent of the available propellants.

b. The heat of reaction is used to adiabatically heat the combustion gases and unreacted propellants to the final equilibrium flame temperature. Heat loss from the fireball occurs via blackbody radiation, and no other heat interchange is expected with the surroundings (i.e., conduction or convection) prior to dispersion by prevailing winds.

c. The reaction is maintained in a "containerless" system, i.e., as gaseous products are formed and the temperature is raised, the cloud volume expands to accommodate the additional hot mass.

d. All combustion products and unreacted propellants are assumed to be uniformly distributed in the final cloud.

e. Any chemical present in higher concentrations than its vapor pressure at the final cloud temperature will condense from the gas cloud and be present as a suspended aerosol, or will fall from the cloud and be deposited at ground level.

f. Reactions will occur in air at one atmosphere nominal pressure, and at ambient humidity levels.

3. Design Goals

The goals for the design of the combustion chamber, the execution of the propellant tests, and the analyses of the reaction products were:

a. Tests should be performed at or near one atmosphere total pressure for the duration of the combustion process;

b. Heat losses to the environment should be minimized (the environment is defined as the system external to the combustion cloud);

c. Tests should be performed at the prevailing environmental conditions of temperature (T) and pressure (P). These parameters should be measured prior to initiating tests;

d. Combustion chamber and propellant delivery systems shall be designed to provide 100 percent ignition and reaction of propellants. This would ensure that results obtained from propellant combustion tests occur from the amounts and types of propellant reacted in the test chamber, and not from the design of the delivery system. Tests under fuel-rich and oxidizer-rich conditions, in which unreacted, vaporized propellant species are expected, shall also be conducted;

e. Propellant quantities (as chemical reactants) shall be delivered in precise, measurable quantities.

f. Chamber contents shall be mixed to ensure homogeneity of gases prior to chemical sampling;

g. Chemical analysis methodology shall be selected to determine composition of target chemicals (mole fraction) from the test chamber at the prevailing chamber conditions (T,P) at which the sampling occurs. Qualitative identification of other species in the gas or condensed phase may be made after the chamber cools;

h. Contamination sources that could contribute to the false identification of chemicals as propellant combustion products shall be eliminated as much as possible during the design, fabrication, cleaning, and analytical portions of the test program. Materials that may artificially introduce chemical contaminants into the test chamber include greases and oils, fingerprints, cleaning solutions (detergents and solvents), rubbers or plastics that may outgas at the expected chamber temperature, and condensed or absorbed water;

i. Chamber surface interactions with reacting propellants or their combustion products shall be minimized. The chamber shall be fabricated from a propellant compatible material (304L stainless steel) and will be preconditioned with Aerozine-50 or dinitrogen tetroxide prior to performing fuel-rich or oxidizer-rich test conditions, respectively, to minimize these interactions; and

j. A baseline analysis of gas composition and condensed residue shall be performed before each test to verify system cleanliness. The chamber shall be decontaminated between tests.

SECTION II

APPROACH

A. DELIVERY SYSTEM DESIGN

The design of the delivery system for liquid rocket propellants employed very coherent, non divergent streams of propellants, which were formed by expelling the liquid contained in a feed reservoir through a square-edged orifice using nitrogen as a pressurizing gas. Liquid propellants included Aerozine-50 (A-50), dinitrogen tetroxide (N_2O_4), Rocket Propellant 1 (RP-1), and gaseous oxygen (GOX). Proper selection of orifice diameters and nitrogen drive pressures enabled the selection of precise metering of propellant mass flowrates and the amounts of propellants introduced into the chamber. Propellant flowrate calculations are contained in Appendix A. Chemical recovery tests were also performed. Unreacted propellants (Aerozine-50 and dinitrogen tetroxide) and expected combustion gases (carbon monoxide and carbon dioxide) were introduced into the chamber, sampled, and analyzed to demonstrate the validity of the chemical sampling and analysis methods.

For the Titan IV liquid propellants (Aerozine-50 and dinitrogen tetroxide), the two propellant streams were aligned so that an intersection of the two paths was achieved 2 to 3 inches from the chamber wall. At the intersection point, the two streams resulted in hypergolic ignition and did not require any external ignition source.

For the Delta II liquid propellants (RP-1 and gaseous oxygen), the gaseous oxygen stream was used to atomize the RP-1, and a high energy spark ignitor (Auburn I-75-1) was placed in the intersection path of the two streams. This spark ignition source served to ignite and sustain the RP-1 combustion. In addition, because previous reports have indicated a difficulty to achieve ignition of the RP-1 fuel, and because the reported vapor pressure of the RP-1 at room temperature was extremely low¹, the fuel was preheated to 100°C to improve combustion conditions. Flows of the liquid propellants were controlled by a sequencing programming rack which supplied voltage to solenoid operating valves (SOVs) downstream of the propellant reservoirs. Dead volumes between the solenoid valves and the nozzle orifice were kept to a minimum, and were measured to be approximately 1.7 cubic centimeters. Dead volumes were replaced with fresh liquid prior to the performance of each test.

Solid propellants (PBAN and Castor IVA) were carefully cut from raw material blocks and placed in a stainless steel support cage inside the combustion chamber. Propellant ignition was achieved by passing current through a high resistivity (2 ohms per foot) nichrome wire embedded in the propellant sample.

Tests combusting only liquid propellants, liquid propellants along with their solid propellant counterpart, and liquid propellants of different mixture ratios (fuel-rich or oxidant-rich) were performed. The last example was expected to yield salient information on the fate of unreacted liquid propellants during fireball formation.

The test matrix (Table 1) shows the propellant combinations, orifice sizes, and pressure requirements used during this test program.

¹Approximately 13 mm Hg at 24°C

TABLE 1. TEST MATRIX AND OPERATING PARAMETERS
PROPELLANT INTERACTION STUDY

| TEST NUMBER | COMPOUND | QUANTITY | TIME, seconds | FLOW RATE, lb/s | ORIFICE SIZE, in. | ΔP , psig |
|-------------|-------------------------------|---------------------|---------------|-----------------|-------------------|-------------------|
| 0A | CO/CO ₂ | 7.5 L/3.0 L | N/A | N/A | N/A | N/A |
| 0B | A-50 | 4.0 cm ³ | 3 | 0.00264 | 0.0128 | 31 |
| 0C | N ₂ O ₄ | 5.5 cm ³ | 3 | 0.0058 | 0.018 | 33 |
| 1 | N ₂ O ₄ | 1.4 cm ³ | 2 | 0.0022 | 0.0128 | 13 |
| | A-50 | 5.0 cm ³ | 2 | 0.0050 | 0.018 | 39 |
| 2 | N ₂ O ₄ | 6.9 cm ³ | 2 | 0.0109 | 0.018 | 116 |
| | A-50 | 2.5 cm ³ | 2 | 0.0025 | 0.0128 | 27 |
| 3 | N ₂ O ₄ | 6.9 cm ³ | 3 | 0.0073 | 0.018 | 52 |
| | A-50 | 5.0 cm ³ | 3 | 0.0033 | 0.0128 | 48 |
| 4 | N ₂ O ₄ | 6.9 cm ³ | 3 | 0.0073 | 0.018 | 52 |
| | A-50 | 5.0 cm ³ | 3 | 0.0033 | 0.0128 | 48 |
| | PBAN | 10 g | N/A | N/A | N/A | N/A |
| 5 | GOX | 1263 scc | 2 | 0.002 | 0.0312 | 182 |
| | RP-1 | 5.1 cm ³ | 2 | 0.0042 | 0.0135 | 80 |
| 6 | GOX | 6317 scc | 2 | 0.01 | 0.040 | 520 |
| | RP-1 | 2.5 cm ³ | 2 | 0.00082 | 0.0135 | 19 |
| 7 | GOX | 6317 scc | 4 | 0.005 | 0.0312 | 436 |
| | RP-1 | 5.1 cm ³ | 4 | 0.0021 | 0.0135 | 20 |
| 8 | GOX | 6317 scc | 3 | 0.0066 | 0.0312 | 578 |
| | RP-1 | 5.1 cm ³ | 3 | 0.0028 | 0.0135 | 35 |
| | N ₂ O ₄ | 6.9 cm ³ | 3 | 0.0073 | 0.018 | 52 |
| | A-50 | 5.0 cm ³ | 3 | 0.0033 | 0.0128 | 48 |
| | CASTOR IVA | 10 g | N/A | N/A | N/A | N/A |
| 9 | PBAN | 10 g | N/A | N/A | N/A | N/A |
| 10 | CASTOR IVA | 10 g | N/A | N/A | N/A | N/A |

Notes: N/A = Not Applicable
cm³ = cubic centimeters liquid 298 K
scc = standard cubic centimeters gas (273 K, 1 atmosphere)
L = liters gas, ambient T,P
g = grams of solid propellant

B. COMBUSTION CHAMBER

A schematic of the combustion chamber is shown in Appendix B. This chamber incorporates the following features:

1. A 150-liter stainless steel, thermally insulated reaction chamber;
2. A view port for visual or photographic verification of ignition;
3. Aerozine-50/dinitrogen tetroxide pressurized delivery systems, incorporating impinging liquid propellant streams;
4. Ignitor circuits for the PBAN and Castor IVA solid propellants, and for the kerosene (RP-1) liquid propellant;
5. An externally driven, gas-powered fan for the mixing of the combustion gases;
6. 1/2-liter stainless steel bottles for the collection of gas samples;
7. Three types of adsorbent sampling devices:
 - a. Activated charcoal tube for organic vapor collection;
 - b. 2 percent HCl bubbler for hydrazine collection; and
 - c. 0.2 N NaOH bubbler for acid gas collection;
8. A stainless steel witness plate for the collection and analysis of condensates (dimension approximately 8 x 6 inches);
9. A pressure gauge (and transducer) for the measurement of internal chamber pressure;
10. Thermocouples to measure gas and chamber wall temperatures; and
11. A pressure relief valve to protect chamber against overpressures.

Photographs of the combustion chamber are shown in Figures 1 through 3. A photograph of the charcoal tube and glass bubbler is shown in Figure 4.

C. ENGINEERING DATA AND CHAMBER SIZING

The desired size of the combustion chamber was 150 liters (internal volume). This was accomplished by constructing a cylindrical vessel from a 2.0 foot diameter stainless steel pipe section (9.75 inches long) to which two 2:1 elliptical head domes were welded.

1. Propellant Quantities

The chamber size of 150 liters was selected for the combustion of liquid and solid propellants specified for Test 8 (GOX/RP-1, N₂O₄/A-50, Castor IVA). The analytical data presented in Table 2 were used to size the chamber.

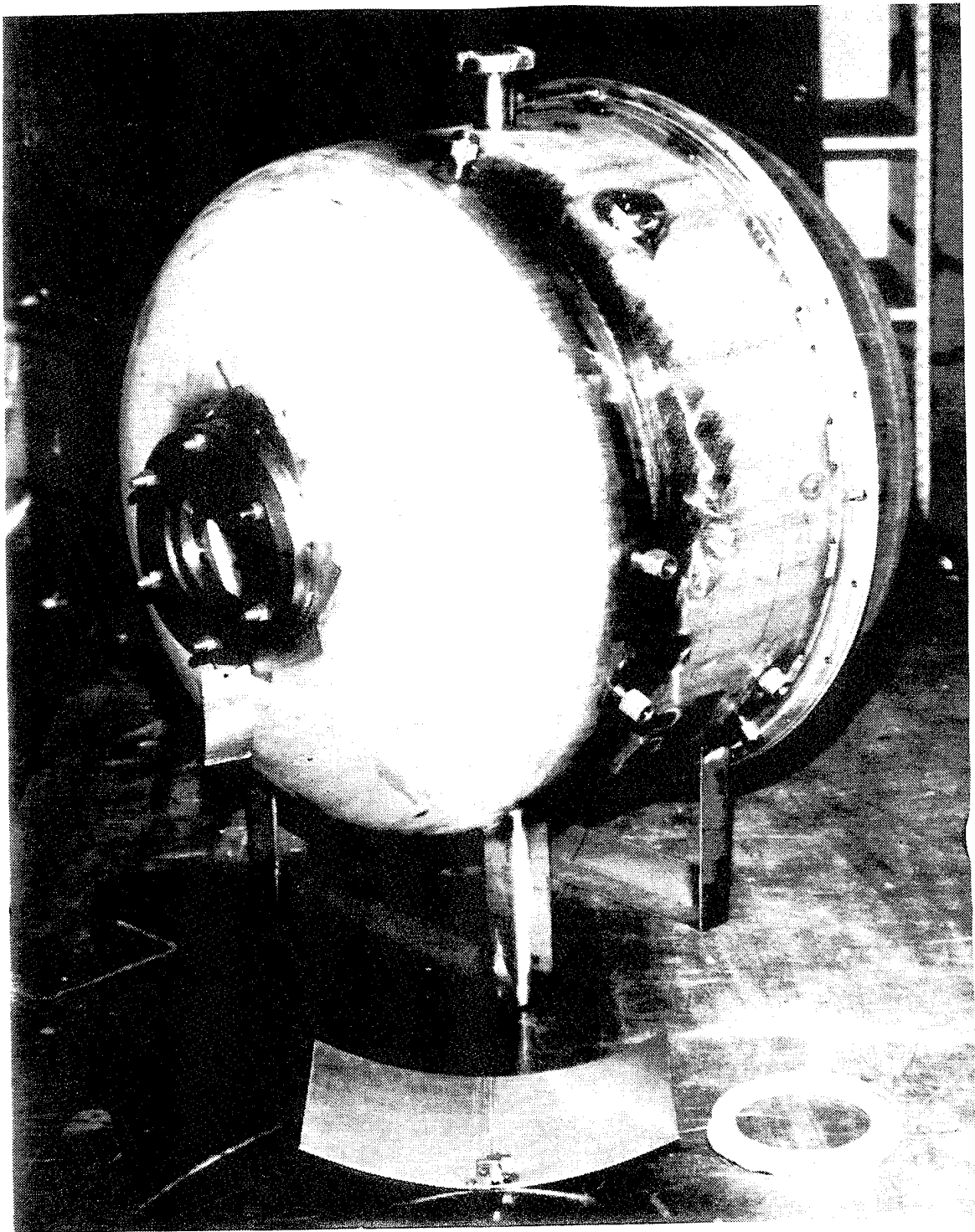


Figure 1. Test Chamber, after Fabrication (Witness Plate and Sight Glass in Foreground).

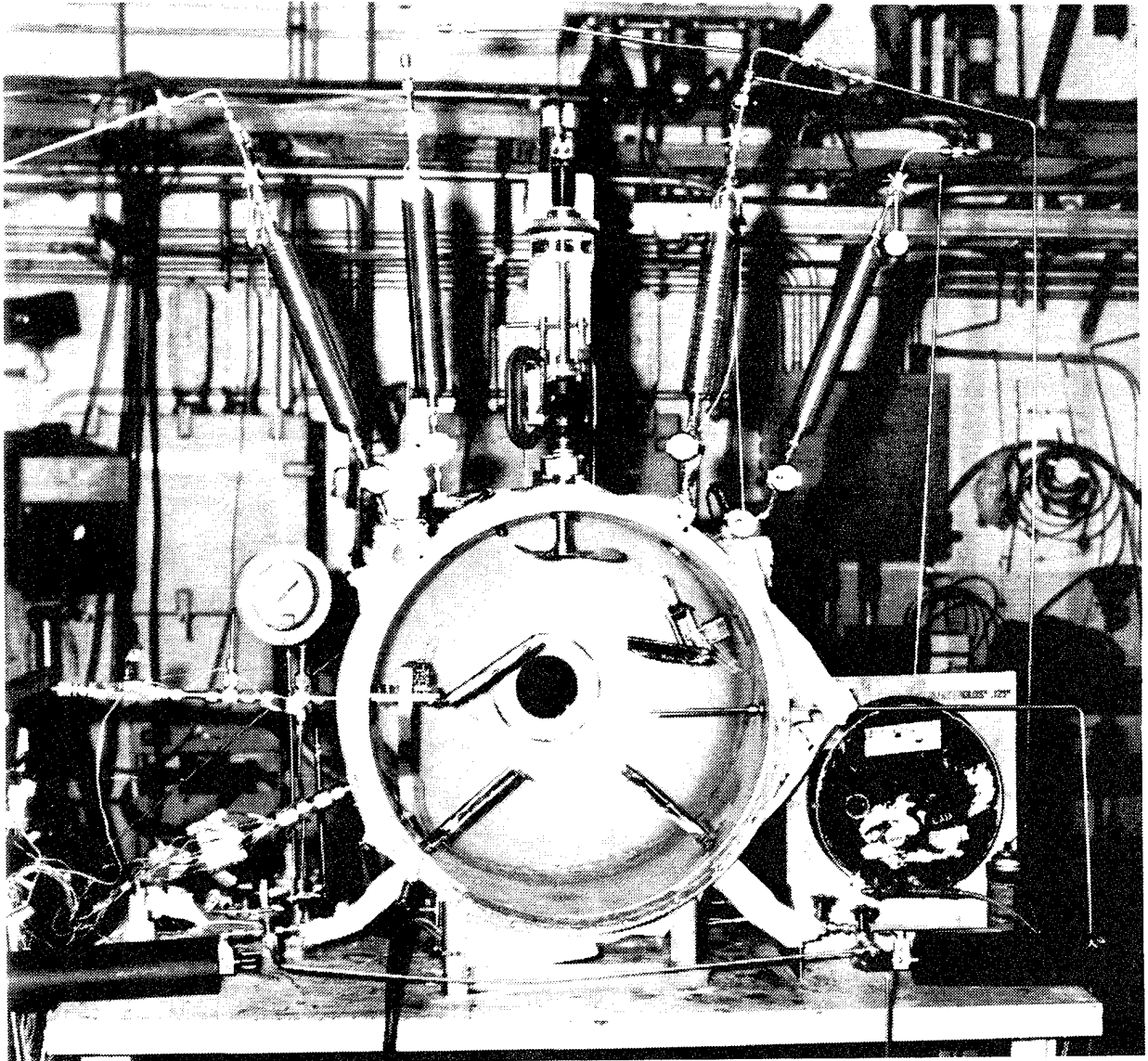


Figure 2. Test Chamber and Support Hardware, Installed at Test Facility.

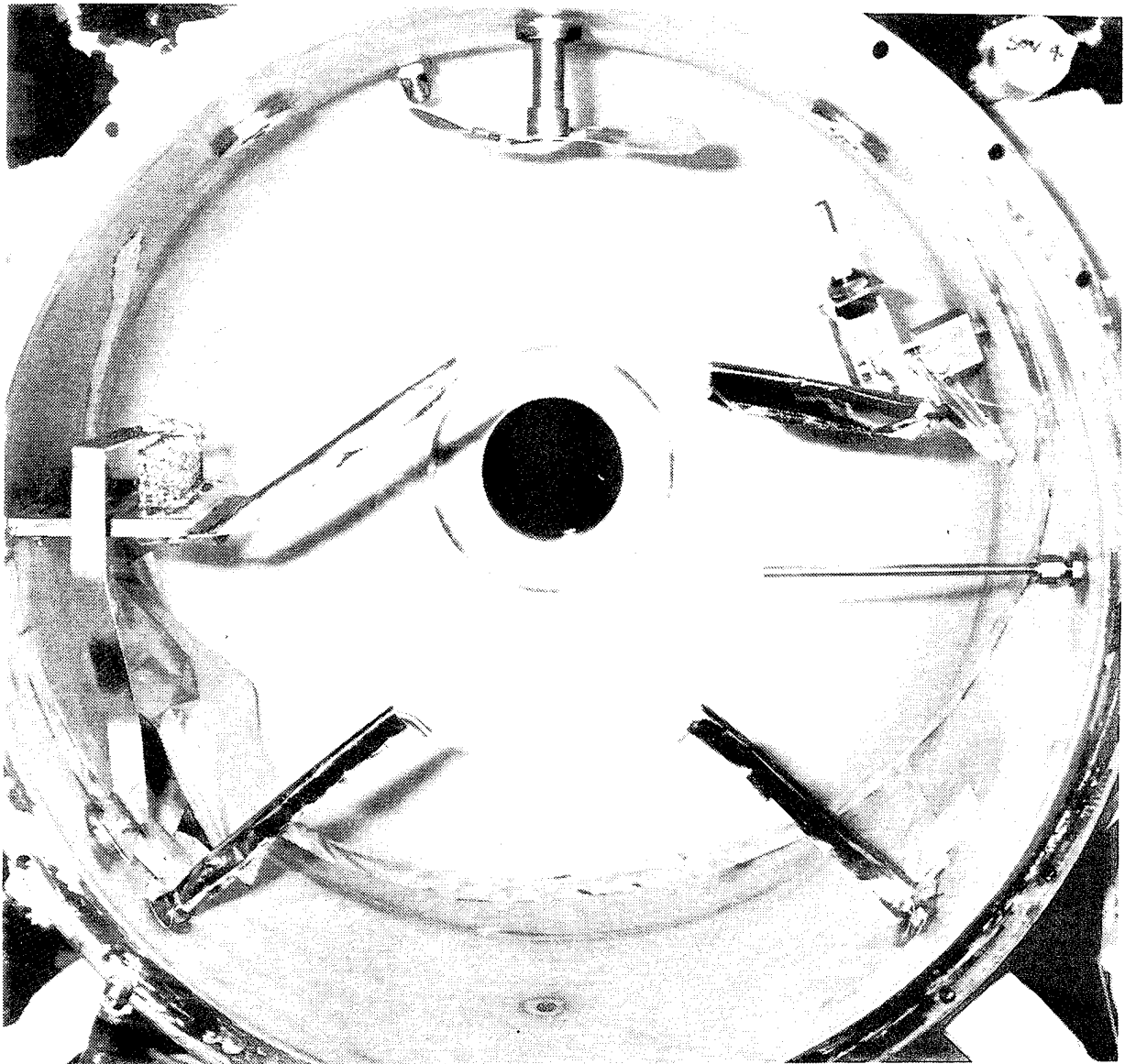


Figure 3. Interior of Test Chamber.

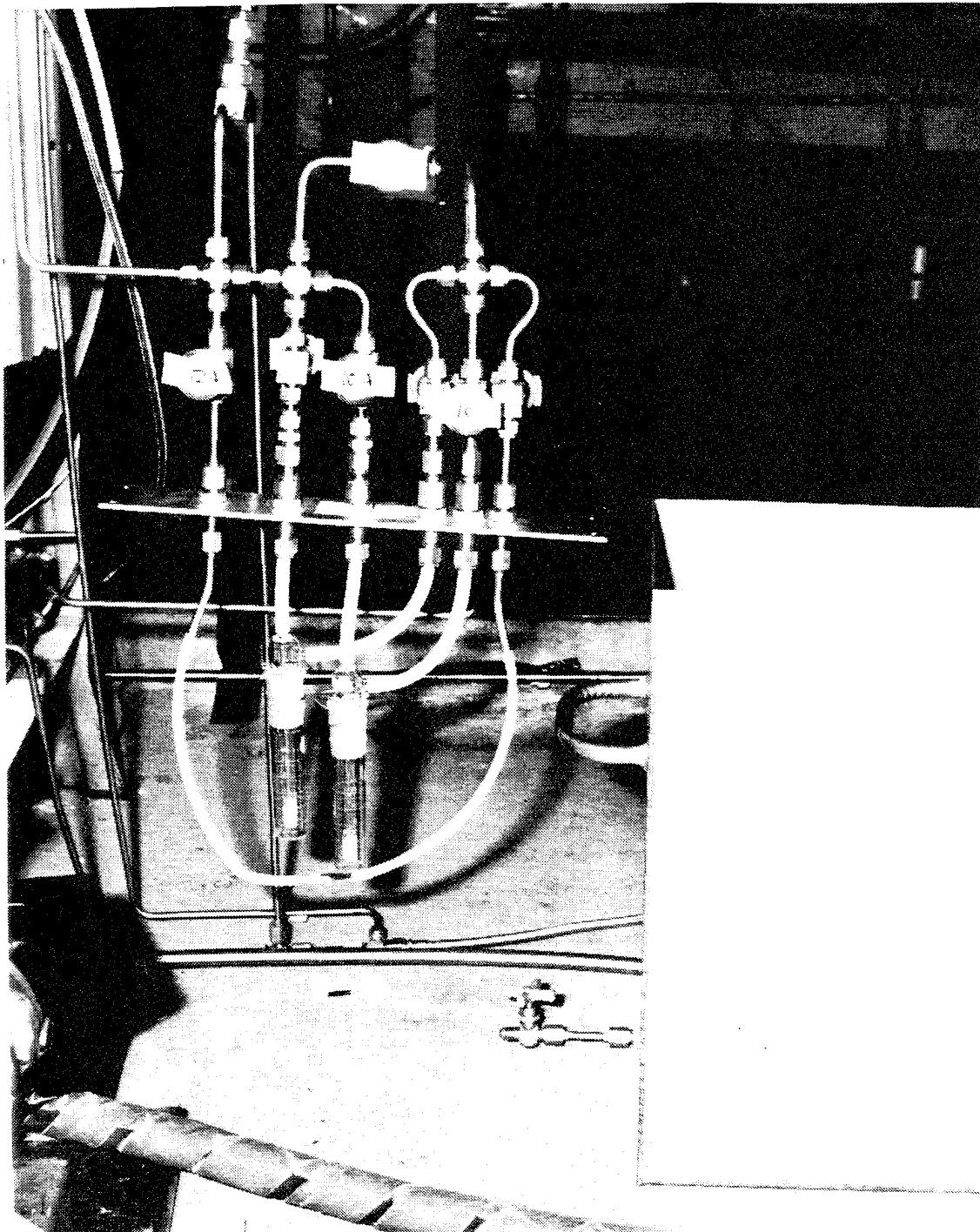


Figure 4. Glass Bubblers and Charcoal Tube.

TABLE 2. AMOUNTS OF PROPELLANTS INTRODUCED AND REACTED IN CHAMBER (TEST 8).

| Propellant | Molecular Weight (grams/mole) | Density (grams per cubic centimeters) | Volume (milliliters) | Mass (grams) | Quantity (moles) |
|-------------------------------|-------------------------------|---------------------------------------|----------------------|--------------|------------------|
| GOX | 32 | 0.001429* | 6320 | 9.04 | 0.282 |
| RP-1 | 170† | ≈0.80 | 5 | 4.0 | 0.024 |
| N ₂ O ₄ | 92 | 1.433 | 6.90 | 9.89 | 0.108 |
| A-50 | 41.8 | 0.899 | 5 | 4.50 | 0.108 |
| Castor IVA | ≈44.9 | 1.705 | N/A | 10.0 | 0.223 |

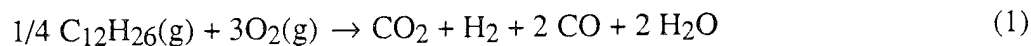
*measured at standard conditions (0°C, 760 mm Hg)

† as n-dodecane, CH₃(CH₂)₁₀CH₃

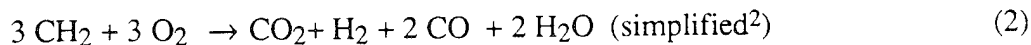
N/A=Not Applicable

2. Propellant Combustion Reactions Expected

a. RP-1 and GOX



or, alternately,

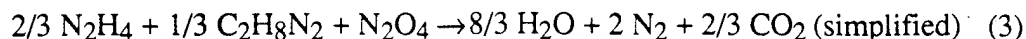


Heat of Combustion = -9.804×10^5 calories/mole n-dodecane (calculated)

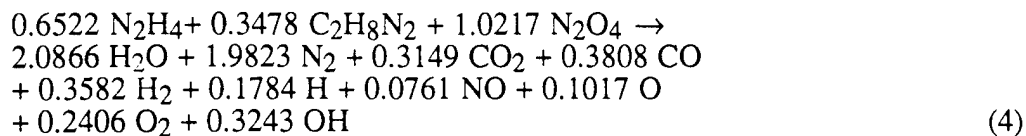
Flame Temperature = 4026 K (calculated)

²CH₂ is the empirical formula for RP-1 kerosene-based rocket fuel. The heat of formation of n-dodecane gas of -69526 calories per mole was used in this analysis in lieu of the value of n-dodecane liquid of -84180 calories per mole because the liquid was preheated to 100°C prior to ignition and combustion.

b. A-50 and N₂O₄

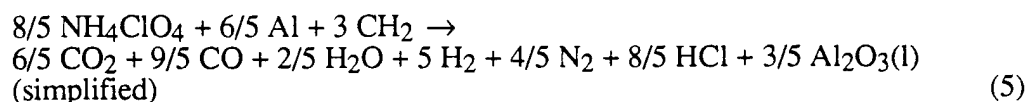


A more complete chemical reaction based on Gibbs Free Energy changes, and used in rocket performance calculations is as follows:



Heat of Reaction = -1.475×10^5 calories/mole N₂O₄ (calculated)
Flame Temperature = 2918 K

c. Castor IVA



The Castor IVA solid rocket propellant consists of a polybutadiene-acrylic acid (PBAA) copolymer. A more complete representation for the combustion products produced by the Castor IVA solid rocket propellant³ is presented in Table 3.

TABLE 3. COMBUSTION PRODUCTS EXPECTED FOR CASTOR IVA SOLID ROCKET PROPELLANT.

| Component | Composition (moles/100 grams) |
|--------------------------------|-------------------------------|
| H ₂ | 1.134 |
| CO | 0.937 |
| HCl | 0.567 |
| H ₂ O | 0.451 |
| N ₂ | 0.295 |
| Al ₂ O ₃ | 0.246 |
| CO ₂ | 0.097 |
| others | 0.004 |

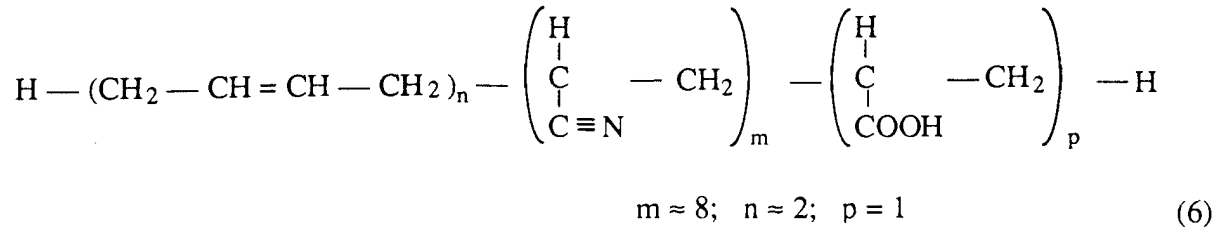
Heat of Reaction = -1.206×10^5 calories/100 grams Castor IVA (calculated)
Flame Temperature = 3246 K (chamber pressure=1000 pounds/square inch)

³CPIA/M2 Propellant Manual. TP-H-8038. Unit 1034. Chemical Propulsion Information Agency. Columbia, Maryland, 1964.

d. PBAN

The Titan IV Solid Rocket Motor (SRM) propellant consists of a terpolymer of polybutadiene-acrylic acid-acrylonitrile (PBAN), the structure of which is shown in Equation (6).

PBAN (approximate structure)



The combustion products expected from ignition of the PBAN are presented in Table 4.

TABLE 4. COMBUSTION PRODUCTS EXPECTED FROM PBAN SOLID ROCKET PROPELLANT.

| Component | Composition (moles/100 grams) |
|--------------------------------|-------------------------------|
| H ₂ | 1.317 |
| CO | 1.077 |
| HCl | 0.572 |
| H ₂ O | 0.323 |
| N ₂ | 0.286 |
| Al ₂ O ₃ | 0.297 |
| CO ₂ | 0.033 |
| others | 0.000 |

Heat of Reaction = -1.322×10^5 calories/100 grams PBAN (calculated)
 Flame Temperature = 3476 K

3. Expected Gaseous Products In Chamber After Combustion

Expected molar quantities of gaseous products resulting from the combustion of solid and liquid propellants in Test 8 are presented in Table 5.

TABLE 5. TOTAL MOLAR INVENTORY OF COMBUSTION GASES EXPECTED IN CHAMBER AFTER PROPELLANT BURNING (TEST 8).

| Compound | GOX/RP-1 (moles) | N ₂ O ₄ /A-50 (moles) | Castor IVA (moles) | Total Moles |
|------------------------------------|------------------|---|--------------------|-------------|
| CO ₂ | 0.094 | 0.0333 | 0.0097 | 0.1370 |
| N ₂ | - | 0.2095 | 0.0295 | 0.2390 |
| H ₂ O | 0.1880 | 0.2206 | 0.0451 | 0.4527 |
| HCl | - | - | 0.0567 | 0.0567 |
| H ₂ | 0.094 | 0.0379 | 0.1134 | 0.2453 |
| Al ₂ O ₃ (s) | - | - | 0.0246† | 0.0246† |
| CO | 0.1880 | 0.0403 | 0.0937 | 0.3220 |
| NO | - | 0.0080 | - | 0.0080 |
| O ₂ | - | 0.0254 | - | 0.0254 |
| OH | - | 0.0343 | - | 0.0343 |
| H | - | 0.0189 | - | 0.0189 |
| O | - | 0.0108 | - | 0.0108 |
| Other | - | - | 0.0004 | 0.0004 |
| Σ | 0.5640 | 0.6390 | 0.3485* | 1.5515* |

*does not include Al₂O₃, a condensate

†not included in total

dashes in table indicate the absence of the particular compound

4. Calculation Of Final Gas Composition In Chamber After Combustion

Table 6 presents the final gas composition expected for Test 8. Note that the composition includes initial amounts of nitrogen and oxygen originally present in the chamber prior to combustion (3.93 moles of N₂ and 1.04 moles of O₂ from 150 liters of gas at 298 K, 0.81 atmospheres pressure). The final expected gas temperature, allowing for heat transfer to the chamber wall, is 304 K. Calculation of this temperature is presented in the following section.

TABLE 6. FINAL EXPECTED GAS COMPOSITION IN CHAMBER (TEST 8).

| Compound | Quantity (moles) | Volume* (liters) | Concentration (percent by volume) |
|-----------------|------------------|------------------|-----------------------------------|
| CO ₂ | 0.1370 | 3.15 | 2.10 |
| N ₂ | 4.1690 | 95.86 | 6.95 |
| HCl | 0.0567 | 1.30 | 0.87 |
| H ₂ | 0.2453 | 5.64 | 3.76 |
| CO | 0.3220 | 7.40 | 4.93 |
| NO | 0.0080 | 0.18 | 0.12 |
| O ₂ | 1.0654 | 24.50 | 16.33 |
| OH† | 0.0343 | 0.79 | 0.53 |
| H† | 0.0189 | 0.43 | 0.29 |
| O† | 0.0108 | 0.25 | 0.17 |
| Other | 0.0004 | 0.01 | 0.01 |
| Σ | 6.5215 | 149.94 | 99.97 |

*298 K, 1.06 atmospheres

†These compounds are thermodynamically stable at expected fireball temperatures (3000 K to 4000 K). As they cool to room temperature, conversion to water vapor, diatomic hydrogen, and diatomic oxygen is expected.

5. Final Gas Temperature, Neglecting Heat Transfer To Walls

$$\text{Heat Released} = Q = [nC_p\Delta T]_{\text{gas}} \quad (7)$$

$$\begin{aligned} &= 9.804 \times 10^5 \text{ calories/mole n-dodecane} \times 0.024 \text{ mole n-dodecane} \\ &+ 1.475 \times 10^5 \text{ calories/mole N}_2\text{O}_4 \times 0.108 \text{ mole N}_2\text{O}_4 \\ &+ 1.206 \times 10^5 \text{ calories/100 grams Castor IVA} \times 10 \text{ grams Castor IVA} \end{aligned}$$

$$\begin{aligned} &= 5.152 \times 10^4 \text{ calories} = nC_p\Delta T \\ &= (6.5215 \text{ moles})(0.2401 \text{ calories/gram-K})(28.75 \text{ grams/mole})\Delta T \end{aligned}$$

$$\begin{aligned} \text{Maximum Temperature Rise} &= \Delta T_{\text{max}} = 1144 \text{ K} \\ \text{Maximum Final Temperature} &= T_F = 1144 + 298 = 1442 \text{ K} \\ \text{For Test 8, the actual } \Delta T_{\text{max}} &\text{ measured was } 621 \text{ K} \end{aligned}$$

6. Equilibrium Temperature Upon Cooling

Assuming 172 pounds of stainless steel chamber are heated by the combustion gases:

$$\text{Heat Absorbed} = Q = [n_{\text{gas}}C_{p\text{gas}} + n_{\text{chamber}}C_{p\text{chamber}}]\Delta T \quad (8)$$

$$\begin{aligned} 5.152 \times 10^4 \text{ calories} &= [6.5215(0.2401)(28.75) + 172 \text{ pounds}(453.6 \text{ grams/pound}) \\ &\times (0.12 \text{ calories/gram-K})]\Delta T \end{aligned}$$

$$\begin{aligned} \text{Equilibrium Temperature Rise Upon Cooling} &= \Delta T_e = 5.5 \text{ K} \\ \text{Equilibrium Temperature Upon Cooling} &= T_e = 298 + 5.5 = 304 \text{ K} \\ \text{For Test 8, the actual } \Delta T_e &\text{ measured was } 8.8 \text{ K.} \end{aligned}$$

7. Maximum Pressure Rise

$$\text{Maximum Pressure Rise} = \Delta P_{\text{max}} = [R/V] \times [n_F T_F - n_i T_i] \quad (9)$$

$$\begin{aligned} &= [(0.08206 \text{ liters-atmospheres/K-mole})/(150 \text{ liters})] \\ &\times [6.5215 \text{ moles} (1442 \text{ K}) - 4.97 \text{ moles} (298 \text{ K})] \end{aligned}$$

$$= 4.33 \text{ atmospheres} = 63.7 \text{ pounds per square inch, gauge}$$

For Test 8, the actual ΔP_{max} measured was 21.9 pounds per square inch, gauge.

8. Equilibrium Pressure Upon Cooling

$$\text{Equilibrium Pressure Upon Cooling} = \Delta P_e = \Delta nRT/V \quad (10)$$

$$= (6.5215 \text{ moles} - 4.97 \text{ moles})(0.082 \text{ liters-atmospheres/K-mole})(298 \text{ K})/150 \text{ liters}$$

$$= 0.25 \text{ atmospheres} = 3.7 \text{ pounds per square inch, gauge}$$

For Test 8, the actual ΔP_e measured was 1.4 pounds per square inch, gauge.

9. The expected gas composition, gas temperatures, and chamber pressures for Tests 1-10 are presented in Table 7.

TABLE 7. PREDICTED REACTANT AND PRODUCT COMPOSITIONS, PROPELLANT INTERACTION TESTS.

| TEST NUMBER | 0A | 0B | 0C | 1* | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
|-----------------------------------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| REACTANTS (moles) | | | | | | | | | | | | | |
| GOX | | | | | | | | 0.0564 | 0.282 | 0.282 | 0.282 | | |
| RP-1 | | | | | | | | 0.024 | 0.0118 | 0.024 | 0.024 | | |
| N2O4 | | | 0.0864 | 0.0216 | 0.108 | 0.108 | 0.108 | | | | 0.108 | | |
| A-50 | | 0.0864 | | 0.108 | 0.053 | 0.108 | 0.108 | | | | 0.108 | | |
| HA SOLIDS | | | | | | | | | | | 0.2227 | | 0.2227 |
| PBAN SOLIDS | | | | | | | 0.211 | | | | | 0.211 | |
| GAS PRODUCTS (moles) | | | | | | | | | | | | | |
| CO2 | 0.130 | | | 0.0540 | 0.0169 | 0.0333 | 0.0366 | 0.0188 | 0.047 | 0.094 | 0.137 | 0.0033 | 0.0097 |
| N2 | | | | 0.0945 | 0.1065 | 0.2095 | 0.2381 | | | | 0.239 | 0.0286 | 0.0295 |
| H2O | | | | 0.1689 | 0.1122 | 0.2206 | 0.2529 | 0.0376 | 0.0941 | 0.188 | 0.4537 | 0.0323 | 0.0451 |
| HCl | | | | | | | 0.0572 | | | | 0.0567 | 0.0572 | 0.0567 |
| H2 | | | | 0.0217 | 0.0192 | 0.0379 | 0.1696 | 0.0188 | 0.047 | 0.094 | 0.2453 | 0.1317 | 0.1134 |
| CO | 0.3260 | | | 0.0081 | 0.0205 | 0.0403 | 0.148 | 0.0376 | 0.0941 | 0.188 | 0.322 | 0.1077 | 0.0937 |
| NO | | | | 0.0017 | 0.0041 | 0.008 | 0.008 | | | | 0.008 | | |
| O2 | | | | 0.0051 | 0.0129 | 0.0254 | 0.0254 | | 0.1408 | | 0.0254 | | |
| OH | | | | 0.0069 | 0.0174 | 0.0343 | 0.0343 | | | | 0.0343 | | |
| H | | | | 0.0039 | 0.0096 | 0.0189 | 0.0189 | | | | 0.0189 | | |
| O | | | | 0.0022 | 0.0055 | 0.0108 | 0.0108 | | | | 0.0108 | | |
| N2H4 | | 0.0564 | | 0.0134 | | | | | | | | | |
| C2H6N2 | | 0.0300 | | 0.0063 | | | | | | | | | |
| NO2 | | | 0.1728 | 0.1073 | | | | | | | | | |
| NH3 | | | | 0.0282 | | | | | | | | | |
| HYDROCARBONS (RP-1) | | | | | | | | 0.0192 | | | | | |
| OTHER | | | | | | | | | | | 0.0004 | | 0.0004 |
| Σ | 0.456 | 0.0864 | 0.1728 | 0.4148 | 0.4321 | 0.639 | 0.9998 | 0.132 | 0.423 | 0.564 | 1.5515 | 0.3608 | 0.3485 |
| CONDENSED PRODUCTS (moles) | | | | | | | | | | | | | |
| Al2O3 | | | | | | | 0.0297 | | | | 0.0246 | 0.0297 | 0.0246 |
| HEAT RELEASE (calories) | | | | | | | | | | | | | |
| ΔPmax, atmospheres | 0.0743 | 0.0141 | 0.0282 | 1.2697 | 0.6169 | 1.3666 | 2.4731 | 0.3867 | 0.9827 | 1.9567 | 4.3359 | 1.1065 | 1.0126 |
| ΔPe, atmospheres | 0.0743 | 0.0141 | 0.0281 | 0.0497 | 0.0704 | 0.1041 | 0.1629 | 0.0215 | 0.0689 | 0.0919 | 0.2527 | 0.0588 | 0.0568 |
| ΔTmax, K | 0 | 0 | 0 | 422.72 | 184.93 | 411.43 | 707.37 | 130.84 | 309.72 | 615.96 | 1144.4 | 359.26 | 328.49 |
| ΔTe, K | 0 | 0 | 0 | 1.6717 | 0.7337 | 1.6946 | 3.1 | 0.4904 | 1.2267 | 2.5031 | 5.4768 | 1.4066 | 1.2832 |

*50% excess Hz monodecomposes; 26.2% excess Hz combusted with air; 23.8% excess Hz vaporizes

78.9% excess UDMH combusts with air; 21.1% excess UDMH vaporizes

Initial Moles Air in Chamber = 4.97 (150 L at 298K, 0.91 atm pressure)

D. TEST METHODS

1. Chamber Preparation And Check Out

Prior to propellant testing, all critical components such as propellant feed lines and delivery systems, the mixing fan, gas sampling and chemical analysis hardware, ignition circuits, and temperature and pressure measurement equipment were tested for proper operation. The mass of the chamber was determined by weighing it on a platform balance. The internal volume of the chamber was determined by filling the chamber with water, recording the weight change due to the water, and dividing this difference by the density of water (2.198 pounds per liter) to determine the filled volume. Proper valve sequencing operations and timing were verified. Liquid flow rates through the delivery nozzles were also measured, using water as the test liquid.

2. Materials

Materials used in this test are listed below.

Dinitrogen tetroxide (MIL-P-26539C, MON-1)

Aerazine-50 (MIL-P-27402A)

RP-1 (MIL-P-25576C)

Gaseous oxygen was obtained commercially in a high pressure cylinder

Castor IVA propellant, as obtained from Thiokol Corporation, formulation TP-H-8299

PBAN propellant, as obtained from United Technologies, formulation UTP-3001B

Liquid propellants were loaded into clean, 0.5-liter stainless steel Hoke® bottles, and sealed under gaseous nitrogen prior to attachment to the test system.

3. Open Chamber Combustion Tests

Twenty-three propellant combustion tests were performed with the chamber dome removed. The purpose of these tests was to visually observe the burning of liquid and solid rocket propellants to verify proper operation of the propellant delivery system. Results of the open chamber tests were recorded using high speed video photography and were used to optimize the combustion process. The decision to conduct these preliminary tests with RP-1/gaseous oxygen, A-50/dinitrogen tetroxide, and the Titan IV PBAN solid propellant proved essential in the primary focus of this study, the development of the sealed chamber combustion tests.

4. Chamber Recovery Tests - Test Series 0

A series of tests was performed to validate the chemical analysis methods and to demonstrate recovery of gases artificially introduced into the chamber. These tests were performed using dry nitrogen as a balance gas (to eliminate reactions with air). Recovery tests were performed with the chamber equilibrated at 50°C. Brief descriptions of the chamber recovery tests are given below.

Test 0A - Carbon monoxide (CO) and carbon dioxide (CO₂) were introduced into the chamber as pure gases, to final volumetric concentrations of 4.5 volume percent and 1.8 volume percent, respectively. Upon completion of the CO and CO₂ addition, the mixing fan was turned on and gas samples were withdrawn from the chamber at selected time intervals (5, 10, and 15 minutes) for chemical analyses. After testing, the chamber was purged with warm (50°C) nitrogen for 1 hour and allowed to cool to room temperature in preparation for the next recovery test.

Test 0B - The recovery of Aerozine-50 from the test chamber was performed after completion of Test 1 "A-50/N₂O₄ Fuel Rich Combustion Test". Prior to testing, the chamber was exposed overnight to 0.0864 moles of A-50 to condition the chemically active sites on the stainless steel surfaces with the propellant. After completing Test 1, an aliquot of Aerozine-50 (0.0864 moles, or 3.61 grams) was introduced into the chamber through the hydrazine nozzle. The mixing fan was turned on and samples were withdrawn from the chamber after 15 and 30 minutes and analyzed. A final gas sample was taken 2 hours after the propellant was introduced into the chamber. After completing the test, the chamber was purged with warm (50°C) nitrogen, wiped with isopropyl alcohol (IPA), rinsed with deionized water and isopropyl alcohol, and purged again with warm nitrogen to dry. The chamber was cooled to room temperature before initiating the next test. A flowchart showing the procedure used to condition the chamber with Aerozine-50, to conduct Test 1, and to conduct the Aerozine-50 recovery test is presented in Figure 5.

Test 0C - The recovery of dinitrogen tetroxide from the chamber was likewise conducted after completing Test 2 "A-50/N₂O₄ Oxidizer Rich Combustion". For these tests, the chamber was preconditioned with 0.0864 moles of dinitrogen tetroxide overnight. After completing Test 2, an aliquot of dinitrogen tetroxide (0.0864 moles, or 7.95 grams) was introduced into the nitrogen filled chamber through the dinitrogen tetroxide nozzle. The mixing fan was turned on, and samples were taken and analyzed after 15 and 30 minutes. After completing the test, the chamber was wiped with isopropyl alcohol, rinsed with water and isopropyl alcohol, purged with warm (50°C) nitrogen for 1 hour, and cooled to room temperature. A flowchart showing the procedure used to condition the chamber with dinitrogen tetroxide, to conduct Test 2, and to conduct the dinitrogen tetroxide recovery test is presented in Figure 6.

5. Propellant Interaction Tests

The remainder of the test series (Test 3 through Test 10) were conducted with the propellant types and quantities presented in Table 1. A flowchart for the general procedure used to conduct these tests is presented in Figure 7. Prior to each test, nozzles were carefully aligned, stream impingement was verified using water, and after drying, dead volumes in the nozzles were filled with fresh propellant. For RP-1/GOX combustion, the RP-1 was preheated to 100°C prior to injection into the chamber to improve its combustion properties. Before initiating combustion, the contents of the chamber were purged with bottled breathing air, and gas samples were withdrawn from the chamber to serve as a sample blank. After removal of sample blanks from the chamber, air was reintroduced into the chamber to a final pressure of 0.81 atmospheres (0 pounds per square inch, gauge), liquid delivery pressures were established, valve actuation times were set, and data acquisition (temperature and pressure) begun. Propellant mixing and combustion were accomplished remotely from the test cell, by the use of a sequencing console, which was used to send the electrical actuation signals to the solenoid operating valves. Ignition of the propellants was observed through the view port of the combustion chamber and was recorded by a video camera. After combustion of the propellants was complete, the mixing fan was turned on and samples were taken after 15 and 30 minutes elapsed time. After the last sample was taken, data acquisition was terminated and the test was complete. Gas pressures in the chamber and in the propellant feed lines were vented to an exhaust duct at this time. Wearing protective clothing and breathing apparatus, the operator opened the chamber door, removed the witness plate, and sealed it in a plastic bag. The internal surfaces of the chamber were then cleaned with isopropyl alcohol and water, and dried with a warm nitrogen purge. The gas samples, adsorbent tubes, bubblers, and witness plate were transported to the laboratory for chemical analysis.

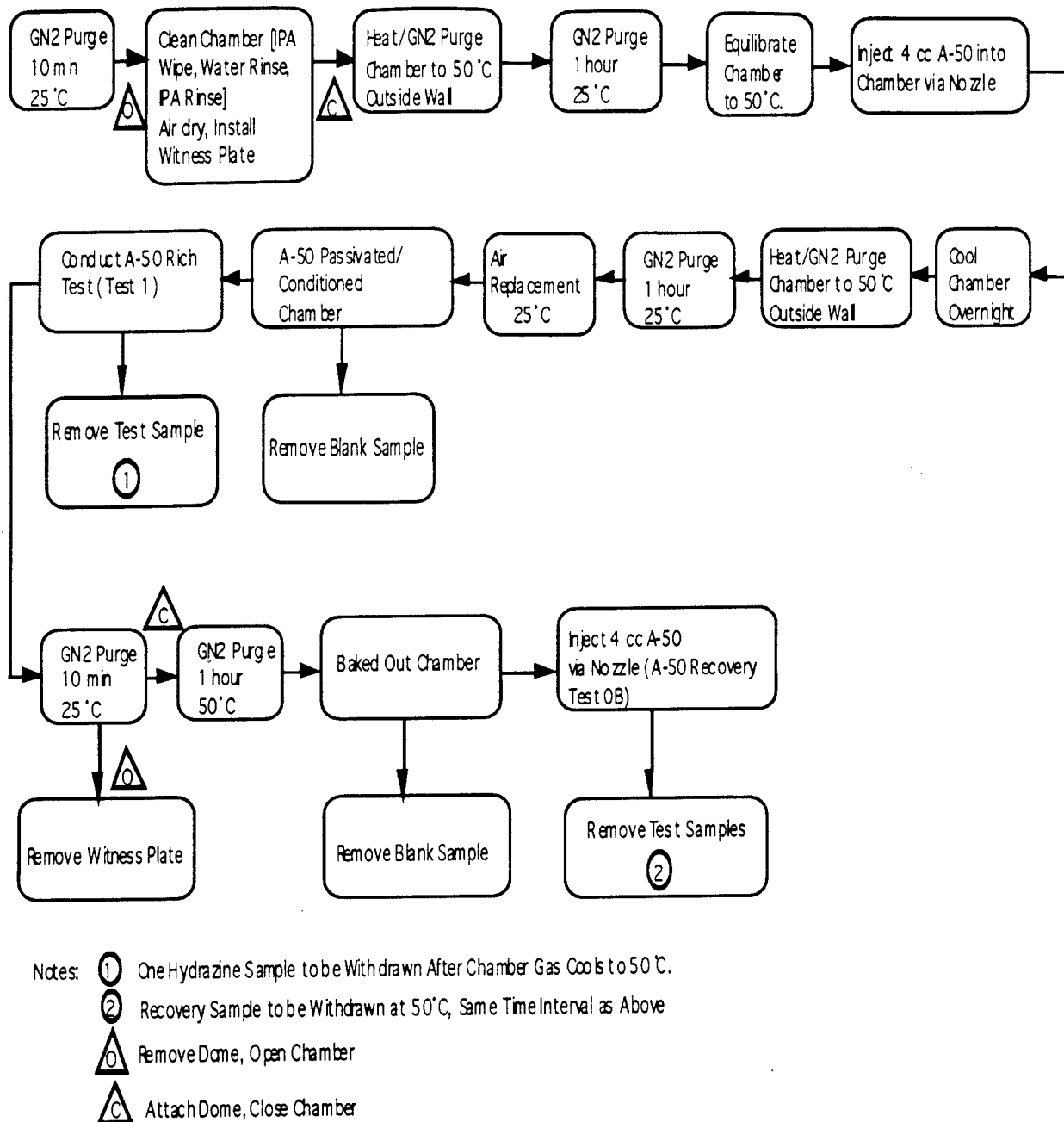


Figure 5. Flowchart, A-50 Chamber Conditioning, A-50 Rich Test, A-50 Recovery Test.

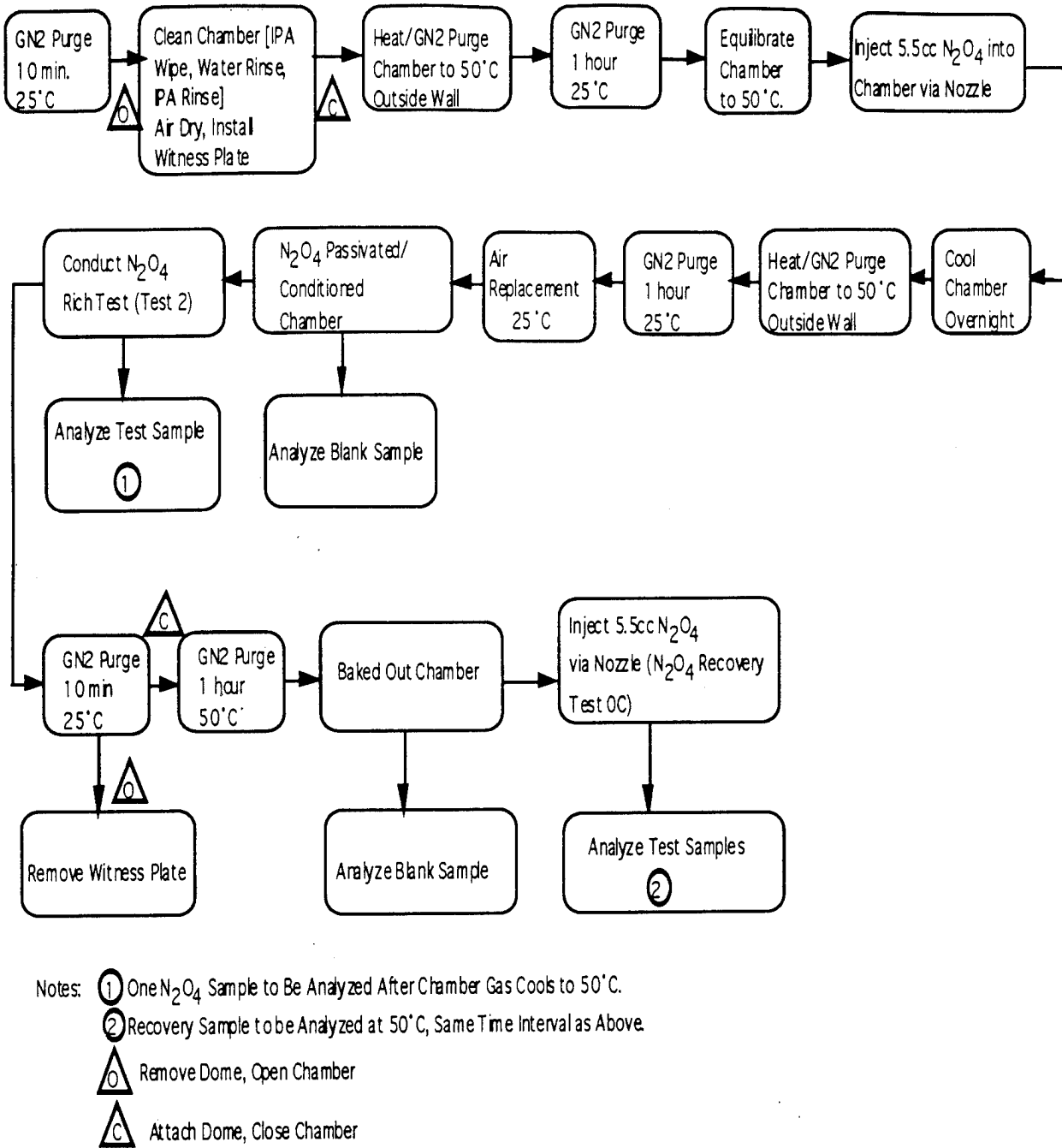
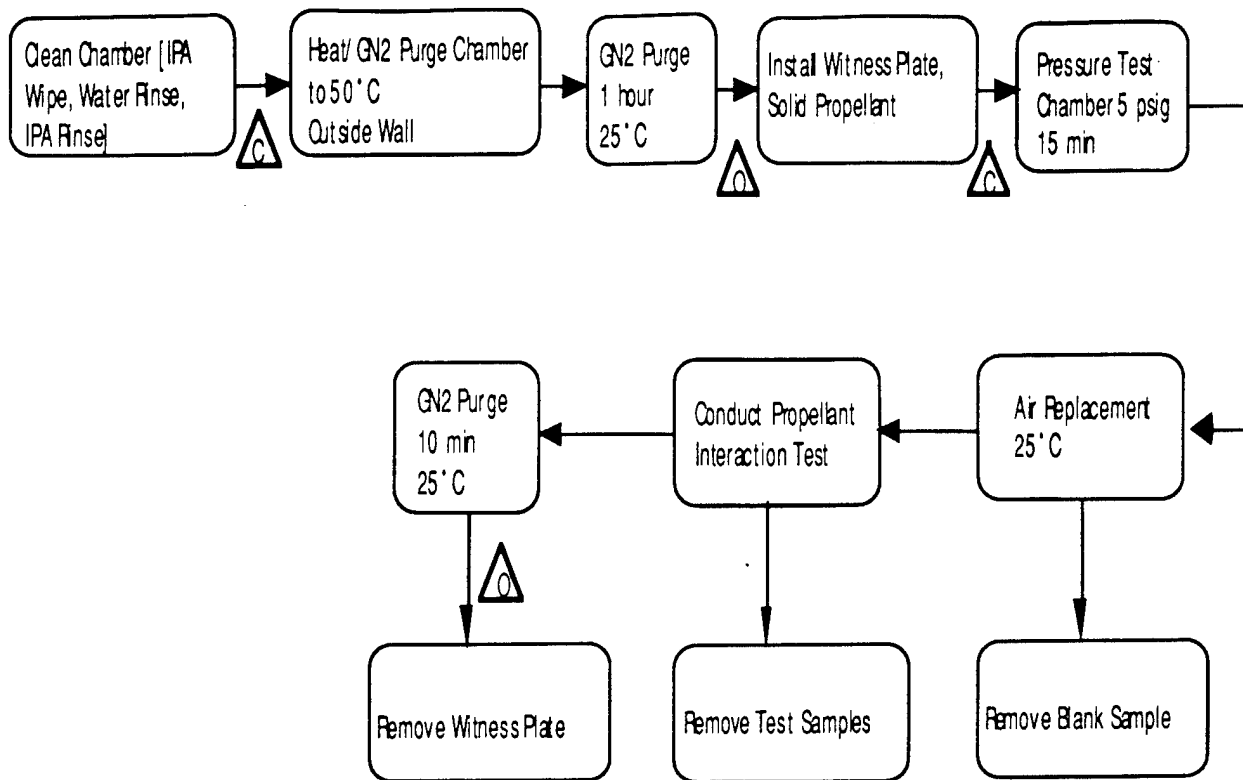


Figure 6. Flowchart, N_2O_4 Chamber Conditioning, N_2O_4 Rich Test, N_2O_4 Recovery Test.



Notes:  Remove Dome, Open Chamber


 Attach Dome, Close Chamber

Figure 7. Flowchart, Propellant Interaction Tests.

E. CHEMICAL ANALYSES

1. Chamber Gas Samples

Gas samples were removed from the chamber by means of an evacuated 0.5-liter stainless steel Hoke® bottle and transported to the laboratory for chemical analysis. Samples were analyzed on a Varian® 6000 gas chromatograph for carbon monoxide, carbon dioxide, methane, ammonia, hydrogen, and oxygen, according to the analysis matrix presented in Table 8. The instrument was calibrated with appropriate standards prior to each analysis.

2. Aerozine-50 Analysis

The chamber gas was bubbled through 2 percent hydrochloric acid at a flow rate of 350 cubic centimeters per minute (760 mm Hg, 294 K) using a regulated sampling pump. After 3 minutes of sampling, the sampling pump was turned off, the bubbler removed, and the sample was transported to the laboratory for chemical analysis. The contents of the bubbler were reacted with 2-furaldehyde to form furaldehyde azine and furaldehyde dimethylhydrazone, the condensation products of hydrazine and unsymmetrical dimethylhydrazine, respectively. These derivatives were then extracted into ethyl acetate and analyzed on a Varian® 6000 gas chromatograph using a thermionic specific detector. Results were calculated using a calibration curve prepared by using pure stock material.

3. Dinitrogen Tetroxide Analysis

The contents of the chamber were sampled directly into a Thermoelectron® chemiluminescence analyzer and analyzed for nitrogen dioxide (NO₂) and nitric oxide (NO). The instrument was calibrated with primary gas standards prior to each analysis.

4. Hydrogen Chloride Analysis

The chamber gas was bubbled through 0.2N sodium hydroxide at a flow rate of 350 cubic centimeters per minute (760 mm Hg, 294 K) using a regulated sampling pump. After 3 minutes of sampling, the sampling pump was turned off, the bubbler removed, and the sample was transported to the laboratory for chemical analysis. The contents of the bubbler were analyzed for chloride by titrating with a standard solution of silver nitrate.

5. Gas-Phase Organics and Witness Plate Evaluation

The chamber gas was passed through a charcoal tube at a flow rate of 350 cubic centimeters per minute (760 mm Hg, 294 K) using a regulated sampling pump. After 3 minutes of sampling, the sampling pump was turned off, the charcoal tube removed, and the tube was transported to the laboratory for chemical analysis. The tube was extracted with carbon disulfide and analyzed for organic composition using a Finnegan® INCOS-50 gas chromatograph/mass spectrometer.

After removing the witness plate from the chamber and transporting it to the laboratory for analysis, one half of the witness plate was rinsed with methylene chloride and analyzed using a Finnegan® INCOS-50 gas chromatograph/mass spectrometer and a Nicolet® Fourier Transform Infrared (FTIR) spectrometer for organic condensates. The other half of the witness plate was analyzed for pH, using a pH meter or pH indicator paper, and for nitrate by spectrophotometry.

Mass spectra of organic contaminants were compared electronically to reference spectra maintained in an organic chemical library resident on the instrument, and the top five matches were reported. Further details of the analysis methodology can be found in Appendix D.

TABLE 8. CHEMICAL ANALYSES MATRIX, PROPELLANT INTERACTION TESTS.

| TEST NUMBER | 0A | 0B | 0C | 1* | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
|---------------------------------|-----------------|---------------|---------------|--------------------|------------------------|-------------------------|----------------------|--------------------|------------------------|-------------------------|----------------------|-------|--------|
| CONDITION | CO/CO2 recovery | A-50 recovery | N2O4 recovery | A50/N2O4 fuel rich | A50/N2O4 oxidizer rich | A50/N2O4 stoichiometric | Titan IV Propellants | RP-1/GOX fuel rich | RP-1/GOX oxidizer rich | RP-1/GOX stoichiometric | Delta II Propellants | PBAN | CASTOR |
| REACTANTS (moles) | | | | | | | | | | | | | |
| GOX | | | | | | | | 0.0564 | 0.282 | 0.282 | 0.282 | | |
| RP-1 | | | | | | | | 0.024 | 0.01176 | 0.024 | 0.024 | | |
| N2O4 | | | 0.0864 | 0.0216 | 0.108 | 0.108 | 0.108 | | | | 0.108 | | |
| A-50 | | 0.0864 | | 0.108 | 0.053 | 0.108 | 0.108 | | | | 0.108 | | |
| CASTOR IVA SOLIDS | | | | | | | | | | | 0.2227 | | |
| PBAN SOLIDS | | | | | | | 0.211 | | | | 0.2227 | 0.211 | 0.2227 |
| TARGET SPECIES | | | | | | | | | | | | | |
| Carbon Monoxide | X | | | X | X | | X | X | X | X | X | X | X |
| Carbon Dioxide | X | | | X | X | | X | X | X | X | X | X | X |
| Methane | | | | X | X | | X | X | X | X | X | X | X |
| Ammonia | | | | X | | X | | | | | | | |
| Hydrogen | | | | X | X | | X | X | X | X | | X | X |
| Oxygen | | | | X | X | | X | X | X | X | | X | X |
| Hydrazine | | X | | X | | | X | | | | | | |
| Unsymmetrical Dimethylhydrazine | | X | | X | | | X | | | | | | |
| Nitrogen Dioxide | | | X | X | X | X | X | | | | | | |
| Nitric Oxide | | | | X | X | X | X | | | | | | |
| Hydrogen Chloride | | | | | | | X | | | | X | X | X |
| Nitric Acid | | | | | X | | X | | | | X | X | X |
| Gas Phase Organics | | | | X | X | | X | X | | | X | X | X |
| Condensed Organics | | | | X | X | | X | X | X | X | X | X | X |
| pH | | | | X | X | | X | X | X | X | X | X | X |

SECTION III
RESULTS

A. PRETEST MEASUREMENTS

1. Nozzle Flow Rate Calibration And Verification

Nozzle performance test results using water as a test fluid are presented in Table 9. These results were used to establish propellant pressurization requirements using the nozzle-flow equations presented in Appendix C.

TABLE 9. NOZZLE PERFORMANCE TEST RESULTS.

| Nozzle Type | N ₂ O ₄ | N ₂ O ₄ | A-50 | GOX | RP-1 |
|---|-------------------------------|-------------------------------|--------|--------|--------|
| Diameter (inches) | 0.0128 | 0.018 | 0.0128 | 0.0312 | 0.0135 |
| Expulsion Pressure (pounds per square inch, gauge) | 50.0 | 51.0 | 50.0 | 57.0 | 58.9 |
| H ₂ O Flow Rate (milliliters per second) | 1.615 | 2.75 | 1.631 | 8.714 | 1.904 |
| Discharge Coefficient | 0.74 | 0.63 | 0.75 | 0.63 | 0.72 |

2. Chamber Weight And Internal Volume

The weight and internal volume of the chamber were measured by filling the chamber with deionized water and measuring the weight change. The internal volume was computed by dividing by the density of water at 298 K (0.997 grams per cubic centimeter or 2.198 pounds per liter). The results are shown below.

| | |
|-----------------------------|------------------|
| Chamber Weight Empty (Dry) | 172.5 pounds |
| Chamber Weight Water Filled | 513.5 pounds |
| Weight Change | 341.0 pounds |
| Chamber Internal Volume | 155.1 liters |

B. RECOVERY TESTS

1. Carbon Monoxide/Carbon Dioxide Recovery Tests

Results of chamber recovery tests using carbon monoxide and carbon dioxide as tracer gases are shown in Table 10. These materials were introduced into the chamber at target concentrations of 4.5 volume percent and 1.8 volume percent, respectively. CO was recovered at 113 percent of its targeted value, and CO₂ was recovered at 94 percent of its targeted value, after fifteen minutes.

TABLE 10. CARBON MONOXIDE AND CARBON DIOXIDE RECOVERY TEST RESULTS.

| Sample | CO (volume percent) | CO ₂ (volume percent) |
|--------|------------------------|-------------------------------------|
| Blank | ND | ND |
| 5 min | 4.1 | 1.7 |
| 10 min | 4.3 | 1.7 |
| 15 min | 5.1 | 1.7 |

ND = Not Detected

2. Aerozine-50 Recovery Tests

Aerozine-50, 0.0864 moles, was injected into the fuel conditioned chamber at 50°C. This amount corresponds to 1.18 volume percent hydrazine (N₂H₄) and 0.63 volume percent unsymmetrical dimethylhydrazine (C₂H₈N₂) at existing chamber conditions (323 K, 0.806 atmospheres). Results of these chamber recovery tests are given in Table 11. Although there are some fluctuations in the reported results, at the completion of 2 hours, approximately 37 percent of the hydrazine and 108 percent of the dimethylhydrazine were recovered. The low recovery of the hydrazine is not understood, but may be due to absorptions and interactions on the chamber wall or condensation within the chamber. Clearly, this result should be investigated further.

TABLE 11. AEROZINE-50 RECOVERY TEST RESULTS.

| SAMPLE | N ₂ H ₄ (volume percent) | C ₂ H ₈ N ₂ (volume percent) |
|--------------------|---|--|
| Blank | ND | ND |
| 14 minutes | 0.28 | 0.91 |
| 37 minutes | 0.64 | 1.27 |
| 2 hours, 7 minutes | 0.44 | 0.68 |

ND = Not Detected

3. Dinitrogen Tetroxide Recovery Tests

Dinitrogen tetroxide, 0.0864 moles, was injected into the oxidizer-conditioned chamber at 50°C. This corresponded to 0.1728 moles of NO₂ gas which would result in a final target vapor concentration of 3.53 volume percent NO₂ at prevailing chamber conditions (0.797 atmospheres, 319 K). Recovery test results for dinitrogen tetroxide are presented in Table 12. These results indicate very good recovery (113 percent) of dinitrogen tetroxide introduced into the chamber. They are well within the margin of error of the material introduction and sample analyses processes, estimated at plus or minus 25 percent of true value.

TABLE 12. DINITROGEN TETROXIDE RECOVERY TEST RESULTS.

| SAMPLE | NO ₂ (volume percent) |
|------------|-------------------------------------|
| Blank | 0.005 |
| 15 minutes | 3.995 |
| 37 minutes | 3.994 |

C. CHAMBER COMBUSTION TESTS

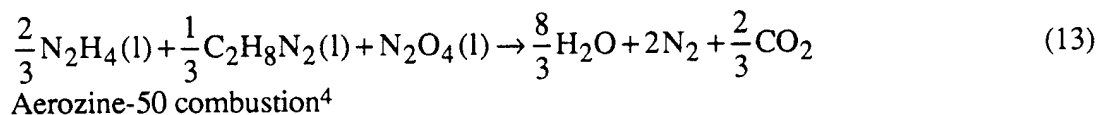
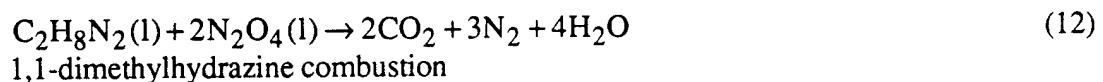
Chemical analyses results for Tests 1 through 10 are presented in Tables 13 through 22, respectively. Because the oxidizer-rich condition for GOX/RP-1 (Test 6) resulted in incomplete burning, very poor results were obtained. This test was repeated and is reported as Test 11 (Table 23).

Temperature and pressure profiles for Tests 1 through 10 are presented in Figures 8 through 27. The temperature and pressure profiles for Test 11 (a repeat of Test 6) are presented in Figures 28 and 29, respectively. Temperatures were measured using four thermocouples placed in the gas space inside the chamber. Pressures were measured by a pressure transducer. Thermocouples and the pressure transducer were calibrated prior to making test measurements.

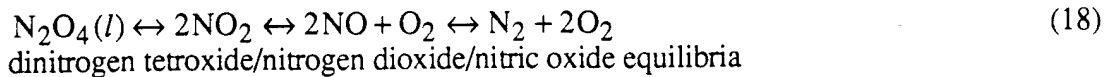
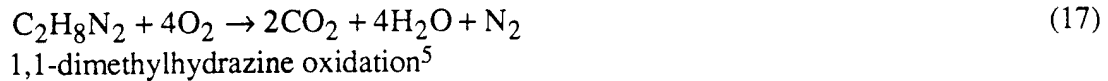
The combustion reactions used to determine the expected compositions in Tables 13 through 23 were based on data contained in Table 7. ΔP_{\max} and ΔT_{\max} were the observed maximum pressure and temperature rises in the chamber, respectively. Expected values of these measurements were calculated using the methods presented in Section II. ΔP_e and ΔT_e were the observed pressure and temperature rises in the chamber upon cooling, after allowing the chamber and combustion gases to attain thermal equilibrium. These data were typically taken 30 minutes after the mixing and combustion of the propellants. Expected values of these measurements were similarly calculated using the methods presented in Section II. These data were also incorporated into Table 7.

Basic chemical reactions for propellants tested in this program are included below:

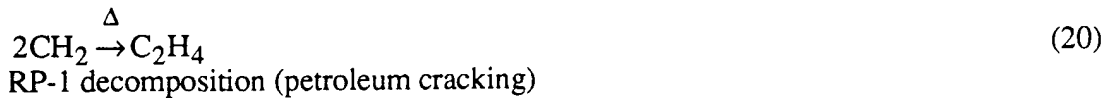
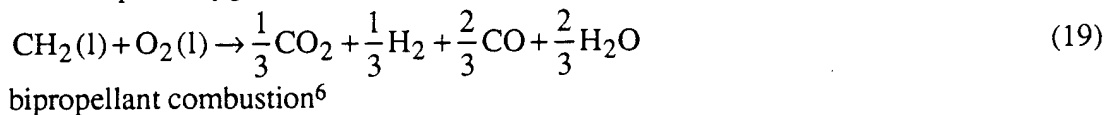
1. Aerozine-50/Dinitrogen Tetroxide



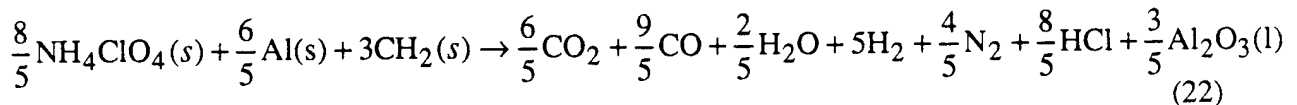
⁴Equation (13) is a simplified reaction equation, not a true representation. Because Aerozine-50 consists of 50 percent hydrazine and 50 percent 1,1-dimethylhydrazine by weight, reacting stoichiometries of these propellants are 0.652 moles N₂H₄, 0.347 moles C₂H₈N₂, and 1.02 moles N₂O₄.



2. RP-1/Liquid Oxygen



3. Solid Rocket Propellant Combustion



⁵Incomplete oxidation products such as formaldehyde dimethylhydrazone (FDH) and nitrosodimethylamine (NDMA) have also been reported in the literature.

⁶CH₂ is the empirical formula for RP-1. It may be approximated by 0.0833 moles of n-dodecane, C₁₂H₂₆.

TABLE 13. RESULTS FROM TEST 1, A-50/N₂O₄ FUEL-RICH COMBUSTION, 5.0 CM³ A-50 (0.108 MOLE); 1.4 CM³ N₂O₄ (0.0216 MOLE).

| Parameter | Blank | Sample 1 | Sample 2 | Expected ** |
|-------------------------------|---------|----------|----------|-------------|
| NH ₃ | ND | ND | ND | 0.52% |
| H ₂ | ND | 0.013%* | 0.013%* | 0.40% |
| O ₂ | 20.0% | 16.7% | 16.5% | 16.9%† |
| CO | ND | 0.01%* | NA | 0.15% |
| CH ₄ | ND | 0.008%* | NA | 0.00% |
| CO ₂ | 0.008%* | 0.82% | NA | 0.99% |
| N ₂ H ₄ | ND | NA | ND | 0.24% |
| UDMH | ND | NA | ND | 0.12% |
| NO ₂ | NA | NA | NA | 0.00% |
| NO | NA | NA | NA | 0.03%‡ |
| HCl | NA | NA | NA | 0.00% |

Comments: Condensed residue, yellow-dark brown in color, was observed in chamber, on witness plate, and on fan blade.

Notes:
 * Response below lowest calibration standard
 ** Based on data in Table 7
 Initial moles of air in chamber = 5.0595 (155 liters at 0.79 atmospheres, 296 K)
 † includes loss of O₂ due to combustion of N₂H₄, UDMH
 ‡ formed at fireball temperatures, NO reacts with air at room temperature to form NO₂
 ND = Not Detected
 NA = Not Analyzed

| Parameter | Blank | Sample 1 |
|---------------------------------|---|--|
| Charcoal Tube | 1,1,4,4-tetramethyl-2-tetrazine 2-ethyl-1-hexanol 2-decen-1-ol Dodecane | Nitrosodimethylamine N-methyl-N-nitrometaneamine 2-ethyl-1-hexanol dodecane 4,7-dimethylundecane |
| Organic Witness Plate: | N,N,N',N' tetramethyl methanedi-amine Nitrosodimethylamine 1-methyl-1,2,4-triazole 4-penten-2-one 2,2'-oxybis ethanol | |
| Inorganic Witness Plate: | pH = 4.5 | |
| ΔP_{max} | observed = 12.4 psig | expected = 18.4 psig |
| ΔP_e | observed = 0.52 psig | expected = 0.71 psig |
| ΔT_{max} | observed = 313 K | expected = 423 K |
| ΔT_e | observed = 6 K | expected = 1.7 K |
| Notes: | By carbon balance, 60% of UDMH converted to CO ₂ vs. 81% predicted | |

TABLE 14. RESULTS FROM TEST 2, A-50/N₂O₄, OXIDIZER-RICH COMBUSTION, 2.5 CM³ A-50 (0.053 MOLES); 6.0 CM³ N₂O₄ (0.108 MOLES).

| Parameter | Blank | Sample 1 | Sample 2 | Expected ** |
|-------------------------------|--------|----------|----------|-------------|
| NH ₃ | NA | NA | NA | 0.00% |
| H ₂ | ND | ND | ND | 0.30% |
| O ₂ | 20.6% | 18.2% | 18.6% | 19.2% |
| CO | ND | ND | 0.01% | 0.37% |
| CH ₄ | 0.002% | 0.002% | 0.003% | 0.00% |
| CO ₂ | ND | 0.58% | 0.53% | 0.31% |
| N ₂ H ₄ | NA | NA | NA | 0.00% |
| UDMH | NA | NA | NA | 0.00% |
| NO ₂ | 3 ppm | 2.84% | 2.85% | 1.94% |
| NO | 2 ppm | 0.062% | 0.047% | 0.07% |
| HCl | NA | NA | NA | 0.00% |

Comments: O/F mole ratio for this test is 2.0 (parameter changed from original O/F = 5.0) to accommodate reliable delivery pressure. Some liquid condensate was observed after opening chamber.

Notes:
 ** Based on data in Table 7
 Initial moles of air in chamber = 5.0949 (155 liters at 0.808 atmospheres, 300 K)
 ppm = parts per million
 O/F = Oxidizer to Fuel
 ND = Not Detected
 NA = Not Analyzed

| Parameter | Blank | Sample 1 |
|-------------------------|---|--------------------------------------|
| Charcoal Tube | None Reported | Nitrosodimethylamine Ethylenimine |
| Organic Witness Plate | N,N,N',N', tetramethyl methanedi-amine 1-methyl-1,2,4-triazole 2-methylpropane Azetidine 3-ethoxy-1-propene | |
| Inorganic Witness Plate | 5,900 µg total nitrate (as HNO ₃) pH = 4.5 | |
| ΔP _{max} | observed = 7.4 psig | expected = 8.9 psig |
| ΔP _e | observed = 0.2 psig | expected = 1.0 psig |
| ΔT _{max} | observed = 90 K | expected = 185 K |
| ΔT _e | observed = 0.9 K | expected = 0.7 K |

Notes: FTIR spectrum of residue removed from witness plate similar to spectrum of ammonium nitrate

TABLE 15. RESULTS FROM TEST 3, A-50/N₂O₄ STOICHIOMETRIC⁷ COMBUSTION, 5.0 CM³ A-50 (0.108 MOLES); 6.9 CM³ N₂O₄ (0.108 MOLES).

| Parameter | Blank | Sample 1 | Sample 2 | Expected ** |
|-------------------------------|--------|----------|----------|-------------|
| NH ₃ | ND | ND | ND | 0.00% |
| H ₂ | NA | NA | NA | 0.66% |
| O ₂ | NA | NA | NA | 19.0% |
| CO | NA | NA | NA | 0.71% |
| CH ₄ | NA | NA | NA | 0.00% |
| CO ₂ | NA | NA | NA | 0.58% |
| N ₂ H ₄ | ND | NA | ND | 0.00% |
| UDMH | ND | NA | ND | 0.00% |
| NO ₂ | 0.014% | 1.708% | 1.689% | 0.00% |
| NO | 15 ppm | 0.062% | 0.051% | 0.14% |
| HCl | NA | NA | NA | 0.00% |

Comments: A large amount of unreacted N₂O₄ was evident. To verify proper function of delivery system nozzles, burn was repeated and recorded with dome removed. A very good combustion resulted, but N₂O₄ was still in excess.

Notes:
 ** Based on data in Table 7
 Initial moles of air in chamber = 5.0145 (155 liters at 0.79 atmospheres, 299 K)
 ppm = parts per million
 ND = Not Detected
 NA = Not Analyzed

| Parameter | Blank | Sample 1 |
|--------------------------|---|----------------------|
| Charcoal Tube | Not Analyzed | Not Analyzed |
| Organic Witness Plate: | Not Analyzed | |
| Inorganic Witness Plate: | 5,600 µg total nitrate (as HNO ₃) pH = 4.5 - 5.0 | |
| ΔP _{max} | observed = 13.9 psig | expected = 19.5 psig |
| ΔP _e | observed = 0.54 psig | expected = 1.48 psig |
| ΔT _{max} | observed = 569 K | expected = 411 K |
| ΔT _e | observed = 3.4 K | expected = 1.7 K |

Notes: 1.7% NO₂ remained unreacted, this is 0.048 moles N₂O₄, or 44% of initial quantity. Hydrazine and UDMH were not detected.

⁷The stoichiometric ratio is not exactly 1:1 by mole, but very close (see reaction equations on previous pages). 1.02 moles of N₂O₄ reacts with one mole of Aerozine-50. Hydrazine and UDMH are in slight excess for this particular test.

TABLE 16. RESULTS FROM TEST 4, A-50/N₂O₄, PBAN SOLIDS,
5.0 CM³ A-50 (0.108 MOLES); 6.9 CM³ N₂O₄ (0.108 MOLES);
10.0 GRAMS PBAN (0.211 MOLES).

| Parameter | Blank | Sample 1 | Sample 2 | Expected ** |
|-------------------------------|--------|----------|----------|-------------|
| NH ₃ | NA | NA | NA | 0.00% |
| H ₂ | ND | ND | ND | 2.79% |
| O ₂ | 20.7% | 13.5% | 13.4% | 17.7% |
| CO | ND | ND | ND | 2.43% |
| CH ₄ | ND | ND | ND | 0.00% |
| CO ₂ | ND | 2.6% | 2.6% | 0.60% |
| N ₂ H ₄ | ND | NA | ND | 0.00% |
| UDMH | ND | NA | ND | 0.00% |
| NO ₂ | 0.014% | 0.992% | 0.856% | 0.00% |
| NO | 2 ppm | 0.178% | 0.104% | 0.13% |
| HCl | ND | 0.049% | NA | 0.94% |

Comments: A very large amount of bronze-colored residue coated the entire upper half of chamber (see photograph in Figure 30). Residue was insoluble in isopropyl alcohol and methylene chloride, but was soluble in water.

Notes:

86% of available carbon (A-50, PBAN) converted to CO₂

** Based on data in Table 7

Initial moles of air in chamber = 5.0755 (155 liters at 0.80 atmospheres, 296 K)

ppm = parts per million

ND = Not Detected

NA = Not Analyzed

| Parameter | Blank | Sample 1 |
|---------------------------------|---|--|
| Charcoal Tube | None reported | Chloroacetonitrile Nitrosodimethylamine 1,2,4,5-tetrazine Chlorobenzene Azidobenzene |
| Organic Witness Plate: | N,N,N',N' tetramethylmethanediamine Nitrosodimethylamine 1,3-diazabicyclohexane 1-methyl-1,2,4 triazole N-acetyl-dl-aspartic acid | |
| Inorganic Witness Plate: | 50,000 µg total nitrate (as HNO ₃) pH = 4.0 | |
| ΔP _{max} | observed = 15.6 psig | expected = 35.8 psig |
| ΔP _e | observed* = 0.47 psig | expected = 2.3 psig |
| ΔT _{max} | observed = 659 K | expected = 707 K |
| ΔT _e | observed* = 8.6 K | expected = 3.1 K |
| Notes: | * P,T measurements taken upon cooling at 15 minutes after ignition | |
| | FTIR spectrum of bronze-colored residue removed from witness plate similar to spectrum of ammonium nitrate | |

TABLE 17. RESULTS FROM TEST 5, RP-1/GOX FUEL-RICH COMBUSTION, 5.1 CM³ RP-1 (0.024 MOLES); 1263 SCC⁸ O₂ (0.0564 MOLES).

| Parameter | Blank | Sample 1 | Sample 2 | Expected ** |
|-------------------------------|--------|----------|----------|-------------|
| NH ₃ | NA | NA | NA | 0.36% |
| H ₂ | ND | ND | ND | 0.36% |
| O ₂ | 19.9% | 14.3% | 14.2% | 19.4% |
| CO | 0.14% | 0.09% | 0.05% | 0.72% |
| CH ₄ | 0.001% | 0.004% | 0.005% | 0.00% |
| CO ₂ | 0.04% | 4.6% | 4.6% | 0.36% |
| N ₂ H ₄ | NA | NA | NA | 0.00% |
| UDMH | NA | NA | NA | 0.00% |
| NO ₂ | NA | NA | NA | 0.00% |
| NO | NA | NA | NA | 0.00% |
| HCl | NA | NA | NA | 0.00% |

Comments: High CO₂ value indicates most all (84%) excess RP-1 reacted with residual air (afterburning).

Notes:
 ** Based on data in Table 7
 Initial moles of air in chamber = 5.0735 (155 liters at 0.79 atmospheres, 294 K)
 ND = Not Detected
 NA = Not Analyzed

| Parameter | Blank | Sample 1 |
|---|---|---|
| Charcoal Tube | Toluene N-methyl-N-nitromethanamine decahydronaphthalene chromatography column bleed | Toluene 4-octan-3-one 2,4-dimethylhexane 1-butylcyclohexane Nonanal |
| Organic Witness Plate: None Reported | | |
| Inorganic Witness Plate: pH = 5.0 | | |
| ΔP_{max} | observed = 18.8 psig | expected = 5.8 psig |
| ΔP_e | observed = 0.5 psig | expected = 0.3 psig |
| ΔT_{max} | observed = 482 K | expected = 131 K |
| ΔT_e | observed = 5.4 K | expected = 0.5 K |
| Notes: | | |

⁸Standard cubic centimeters at 0°C, 760 mm Hg.

TABLE 18. RESULTS FROM TEST 6, RP-1/GOX, OXIDIZER-RICH COMBUSTION, 2.5 CM³ RP-1 (0.0118 MOLES); 6317 SCC O₂ (0.282 MOLES).

| Parameter | Blank | Sample 1 | Sample 2 | Expected ** |
|-------------------------------|---------|----------|----------|-------------|
| NH ₃ | NA | NA | NA | 0.00% |
| H ₂ | ND | ND | ND | 0.84% |
| O ₂ | 19.9% | 26.2%† | 26.2%† | 20.9% |
| CO | 0.12% | 0.10%* | 0.07%* | 1.69% |
| CH ₄ | 0.007%* | 0.004%* | 0.002%* | 0.00% |
| CO ₂ | 0.03%* | 0.04%* | 0.04%* | 0.84% |
| N ₂ H ₄ | NA | NA | NA | 0.00% |
| UDMH | NA | NA | NA | 0.00% |
| NO ₂ | NA | NA | NA | 0.00% |
| NO | NA | NA | NA | 0.00% |
| HCl | NA | NA | NA | 0.00% |

Comments: Extremely poor ignition and combustion was observed visually. This test was repeated (See Table 23).
Less than 5 percent combustion of RP-1 occurred, based on carbon balance.

Notes:
* Response below lowest calibration standard
† Response above highest calibration standard
** Based on data in Table 7
Initial moles of air in chamber = 5.1542 (155 liters at 0.81 atmospheres, 297 K)
ND = Not Detected
NA = Not Analyzed

| Parameter | Blank | Sample 1 |
|--------------------------|----------------------|----------------------|
| Charcoal Tube | Not Analyzed | Not Analyzed |
| Organic Witness Plate: | Not Analyzed | |
| Inorganic Witness Plate: | pH = 5.0 | |
| ΔP_{max} | observed = 3.0 psig | expected = 14.5 psig |
| ΔP_e | observed = 0.94 psig | expected = 0.98 psig |
| ΔT_{max} | observed = 49 K | expected = 310 K |
| ΔT_e | observed = 0.1 K | expected = 1.2 K |
| Notes: | | |

TABLE 19. RESULTS FROM TEST 7, RP-1/GOX, STOICHIOMETRIC COMBUSTION, 5.1 CM³ RP-1 (0.024 MOLES); 6317 SCC O₂ (0.282 MOLES).

| Parameter | Blank | Sample 1 | Sample 2 | Expected ** |
|-------------------------------|---------|----------|----------|-------------|
| NH ₃ | NA | NA | NA | 0.00% |
| H ₂ | ND | 0.004%* | 0.005%* | 1.66% |
| O ₂ | 20.2% | 20.6% | 20.6% | 18.2% |
| CO | 0.09%* | 0.23% | 0.20% | 3.31% |
| CH ₄ | 0.004%* | 0.002%* | 0.002%* | 0.00% |
| CO ₂ | 0.02%* | 2.9% | 2.7% | 1.66% |
| N ₂ H ₄ | NA | NA | NA | 0.00% |
| UDMH | NA | NA | NA | 0.00% |
| NO ₂ | NA | NA | NA | 0.00% |
| NO | NA | NA | NA | 0.00% |
| HCl | NA | NA | NA | 0.00% |

Comments: Results indicate approximately 58% of RP-1 combusted to form carbon dioxide (carbon balance). Burn was visually good.

Notes:
 * Response below lowest calibration standard
 ** Based on data in Table 7
 Initial moles of air in chamber = 5.1099 (155 liters at 0.81 atmospheres, 299 K)
 ND = Not Detected
 NA = Not Analyzed

| Parameter | Blank | Sample 1 |
|--------------------------|----------------------|----------------------|
| Charcoal Tube | Not Analyzed | Not Analyzed |
| Organic Witness Plate: | Not Analyzed | |
| Inorganic Witness Plate: | pH = 5.0 | |
| ΔP_{max} | observed = 14.3 psig | expected = 28.4 psig |
| ΔP_e | observed = 1.2 psig | expected = 1.3 psig |
| ΔT_{max} | observed = 297 K | expected = 616 K |
| ΔT_e | observed = 3.5 K | expected = 2.5 K |
| Notes: | | |

TABLE 20. RESULTS FROM TEST 8, A-50/N₂O₄, RP-1/GOX, CASTOR IVA SOLIDS, 5.0 CM³ A-50 (0.108 MOLES); 6.9 CM³ N₂O₄ (0.108 MOLES); 5.1 CM³ RP-1 (0.024 MOLES); 6317 SCC O₂ (0.282 MOLES); 10 GRAMS CASTOR IVA SOLID PROPELLANT (0.223 MOLES).

| Parameter | Blank | Sample 1 | Sample 2 | Expected ** |
|-------------------------------|----------|----------|----------|-------------|
| NH ₃ | NA | NA | NA | 0.00% |
| H ₂ | NA | NA | NA | 3.59% |
| O ₂ | NA | NA | NA | 16.6% |
| CO | 0.07%* | 0.21% | 0.19% | 4.72% |
| CH ₄ | <0.001%* | 0.003%* | 0.002%* | 0.00% |
| CO ₂ | 0.01%* | 2.5% | 2.4% | 2.01% |
| N ₂ H ₄ | ND | NA | ND | 0.00% |
| UDMH | ND | NA | ND | 0.00% |
| NO ₂ | 22 ppm | 0.268% | 0.188% | 0.00% |
| NO | 10 ppm | 0.112% | 0.082% | 0.12% |
| HCl | ND | ND | NA | 0.83% |

Comments: Visual observation indicated that all propellants ignited.
Conversion of available carbon (UDMH, Castor, RP-1) to CO₂ = 33%

Notes:
* Response below lowest calibration standard
** Based on data in Table 7
Initial moles of air in chamber = 5.2703 (155 liters at 0.80 atmospheres, 288 K)
ppm = parts per million
ND = Not Detected
NA = Not Analyzed

| Parameter | Blank | Sample 1 |
|---------------------------------|---|---|
| Charcoal Tube | 7-methyl-1-undecene 4-methyldecane Undecanal Tridecane 2-ethyl-1-propyl-1,3-propanediyl- 2-methyl propanoate | Nitrosodimethylamine 4,6,8-trimethyl-1-nonene 4-methyl decane decahydronapthalene decahydro-2-methyl napthalene |
| Organic Witness Plate: | Nitrosodimethylamine N,N-dimethylformamide 1-methyl-1,2,4-triazole ethenyl formate 2-pentanol nitrate | |
| Inorganic Witness Plate: | 14,000 µg total nitrate (as HNO ₃) pH = 4.0 - 4.5 | |
| ΔP_{max} | observed = 21.9 psig | expected = 64.7 psig |
| ΔP_e | observed = 1.4 psig | expected = 3.6 psig |
| ΔT_{max} | observed = 621 K | expected = 1144 K |
| ΔT_e | observed = 8.8 K | expected = 5.5 K |
| Notes: | | |

TABLE 21. RESULTS FROM TEST 9, PBAN SOLIDS, 10 GRAMS PBAN (0.211 MOLES).

| Parameter | Blank | Sample 1 | Sample 2 | Expected ** |
|-------------------------------|---------|----------|----------|-------------|
| NH ₃ | NA | NA | NA | 0.00% |
| H ₂ | ND | 0.08%* | 0.08%* | 2.45% |
| O ₂ | 20.1% | 17.7% | 17.7% | 18.7% |
| CO | 0.07%* | 0.06%* | 0.05%* | 2.00% |
| CH ₄ | 0.004%* | 0.003%* | 0.004%* | 0.00% |
| CO ₂ | 0.03%* | 1.8% | 1.9% | 0.06% |
| N ₂ H ₄ | NA | NA | NA | 0.00% |
| UDMH | NA | NA | NA | 0.00% |
| NO ₂ | NA | NA | NA | 0.00% |
| NO | NA | NA | NA | 0.00% |
| HCl | ND | ND | NA | 1.06% |

Comments: White fluffy residue coated internal chamber surface (presumably Al₂O₃).
87% of carbon recovered as CO₂.

Notes:
 * Response below lowest calibration standard
 ** Based on data in Table 7
 Initial moles of air in chamber = 5.009 (155 liters at 0.79 atmospheres, 299 K)
 ND = Not Detected
 NA = Not Analyzed

| Parameter | Blank | Sample 1 |
|--------------------------|----------------------|----------------------|
| Charcoal Tube | Not Analyzed | Not Analyzed |
| Organic Witness Plate: | Not Analyzed | |
| Inorganic Witness Plate: | pH = 4.0 | |
| ΔP_{max} | observed = 12.1 psig | expected = 15.8 psig |
| ΔP_e | observed = 0.4 psig | expected = 0.8 psig |
| ΔT_{max} | observed = 646 K | expected = 359 K |
| ΔT_e | observed = 5.4 K | expected = 1.4 K |
| Notes: | | |

TABLE 22. RESULTS FROM TEST 10, CASTOR IVA SOLIDS,
10 GRAMS CASTOR IVA (0.223 MOLES).

| Parameter | Blank | Sample 1 | Sample 2 | Expected ** |
|-------------------------------|---------|----------|----------|-------------|
| NH ₃ | NA | NA | NA | 0.00% |
| H ₂ | ND | 0.04%* | 0.05%* | 2.03% |
| O ₂ | 19.1% | 17.0% | 16.9% | 17.9% |
| CO | 0.08%* | 0.05%* | 0.05%* | 1.68% |
| CH ₄ | 0.005%* | 0.003%* | 0.003%* | 0.00% |
| CO ₂ | 0.04% | 1.5% | 1.5% | 0.17% |
| N ₂ H ₄ | NA | NA | NA | 0.00% |
| UDMH | NA | NA | NA | 0.00% |
| NO ₂ | NA | NA | NA | 0.00% |
| NO | NA | NA | NA | 0.00% |
| HCl | ND | 0.02% | NA | 1.02% |

Comments: Good combustion visually, apparently not as much heat release as PBAN propellant.
80% of carbon recovered as CO₂.

Notes:
* Response below lowest calibration standard
** Based on data in Table 7
Initial moles of air in chamber = 5.229 (155 liters at 0.80 atmospheres, 290 K)
ND = Not Detected
NA = Not Analyzed

| Parameter | Blank | Sample 1 |
|--------------------------|---------------------|----------------------|
| Charcoal Tube | Not Analyzed | Not Analyzed |
| Organic Witness Plate: | Not Analyzed | |
| Inorganic Witness Plate: | pH = 4.0 - 4.5 | |
| ΔP_{max} | observed = 9.5 psig | expected = 15.4 psig |
| ΔP_e | observed = 0.4 psig | expected = 0.8 psig |
| ΔT_{max} | observed = 370 K | expected = 328 K |
| ΔT_e | observed = 4.2 K | expected = 1.3 K |
| Notes: | | |

TABLE 23. RESULTS FROM TEST 11, RP-1/GOX, OXIDIZER-RICH COMBUSTION, (REPEAT), 2.5 CM³ RP-1 (0.0118 MOLES); 6317 SCC O₂ (0.282 MOLES).

| Parameter | Blank | Sample 1 | Sample 2 | Expected ** |
|-------------------------------|---------|----------|----------|-------------|
| NH ₃ | NA | NA | NA | 0.00% |
| H ₂ | ND | 0.002%* | 0.002%* | 0.84% |
| O ₂ | 19.3% | 25.1% | 25.0% | 20.9% |
| CO | 0.03%* | 0.06%* | 0.06%* | 1.69% |
| CH ₄ | 0.002%* | 0.004%* | 0.004%* | 0.00% |
| CO ₂ | 0.010% | 0.14% | 0.14% | 0.84% |
| N ₂ H ₄ | NA | NA | NA | 0.00% |
| UDMH | NA | NA | NA | 0.00% |
| NO ₂ | NA | NA | NA | 0.00% |
| NO | NA | NA | NA | 0.00% |
| HCl | NA | NA | NA | 0.00% |

Comments: Extremely poor ignition and combustion was observed visually. This test was a repeat of Test 6.

Notes:
 * Response below lowest calibration standard
 ** Based on data in Table 7
 Initial moles of air in chamber = 5.2808 (155 liters at 0.81 atmospheres, 288 K)
 ND = Not Detected
 NA = Not Analyzed

| Parameter | Blank | Sample 1 |
|--------------------------|----------------------|----------------------|
| Charcoal Tube | Not Analyzed | Not Analyzed |
| Organic Witness Plate: | Not Analyzed | |
| Inorganic Witness Plate: | pH = 5.5 | |
| ΔP_{max} | observed = 4.6 psig | expected = 15.1 psig |
| ΔP_e | observed = 0.98 psig | expected = 0.98 psig |
| ΔT_{max} | observed = 70 K | expected = 310 K |
| ΔT_e | observed = 0.78 K | expected = 1.2 K |
| Notes: | | |

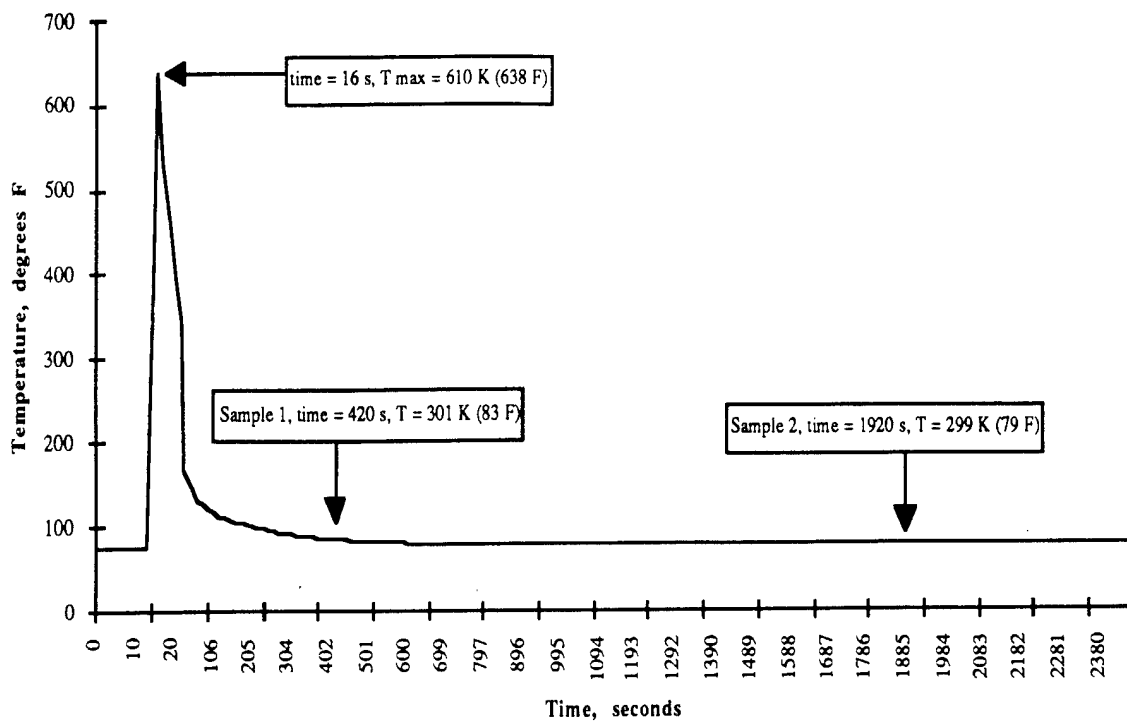


Figure 8. Chamber Gas Temperature, Test 1, A-50/N₂O₄ Fuel-Rich Combustion.

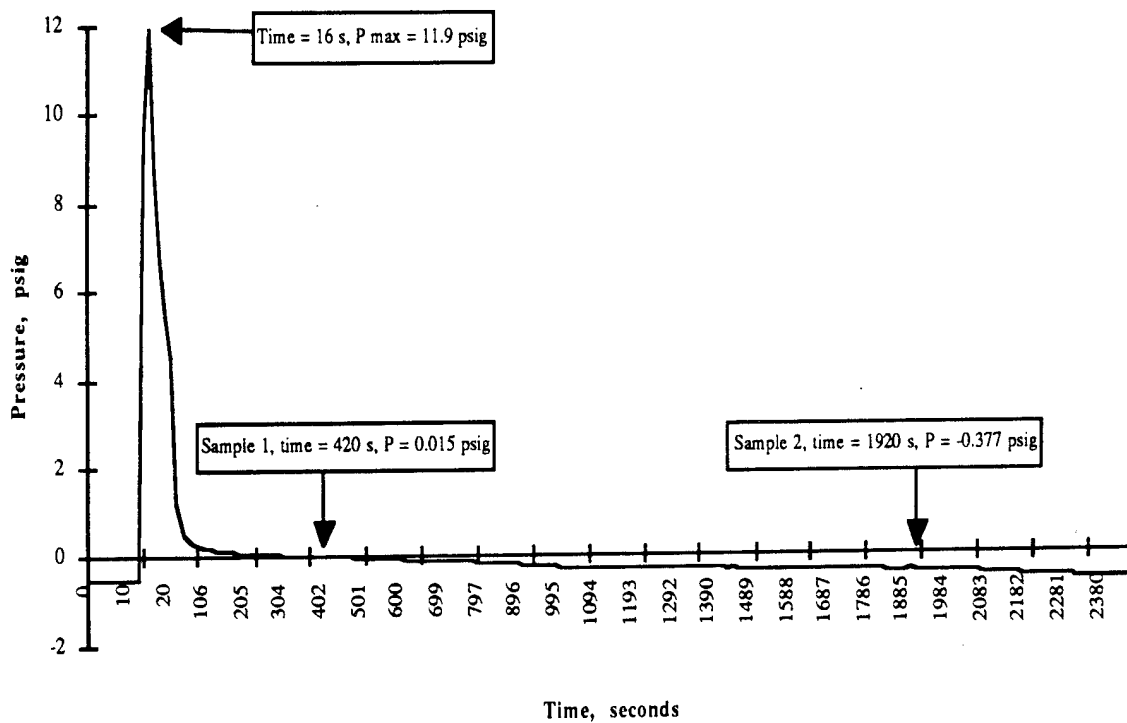


Figure 9. Chamber Pressure, Test 1, A-50/N₂O₄ Fuel-Rich Combustion.

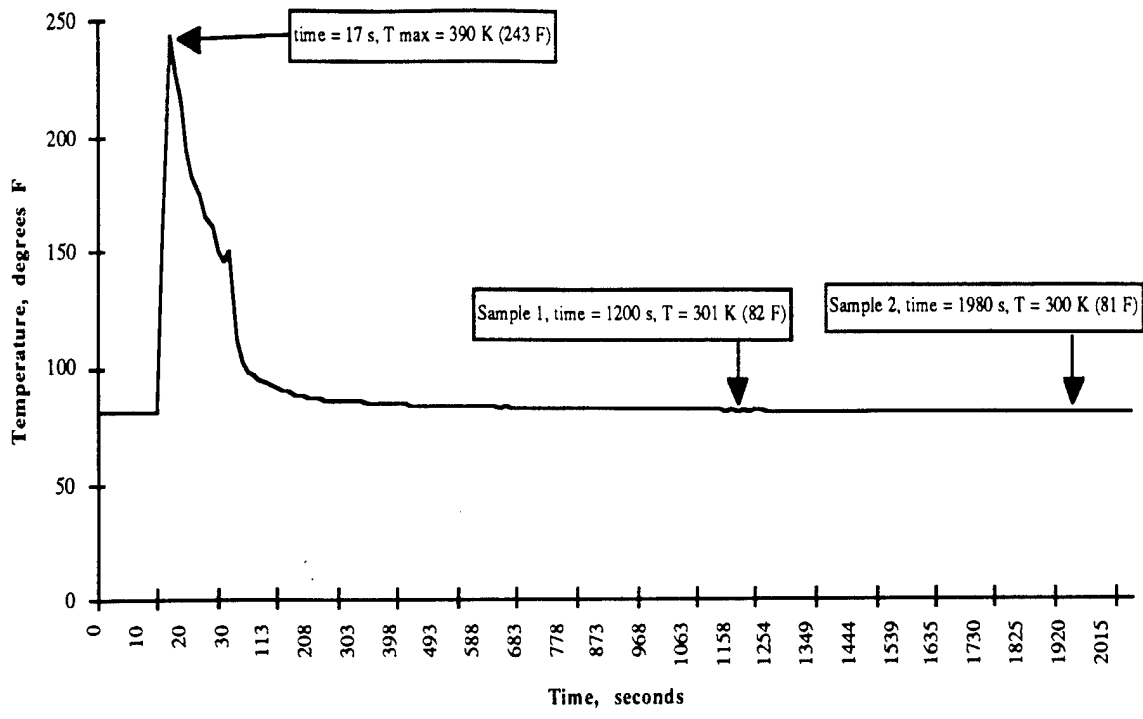


Figure 10. Chamber Gas Temperature, Test 2, A-50/N₂O₄ Oxidizer-Rich Combustion.

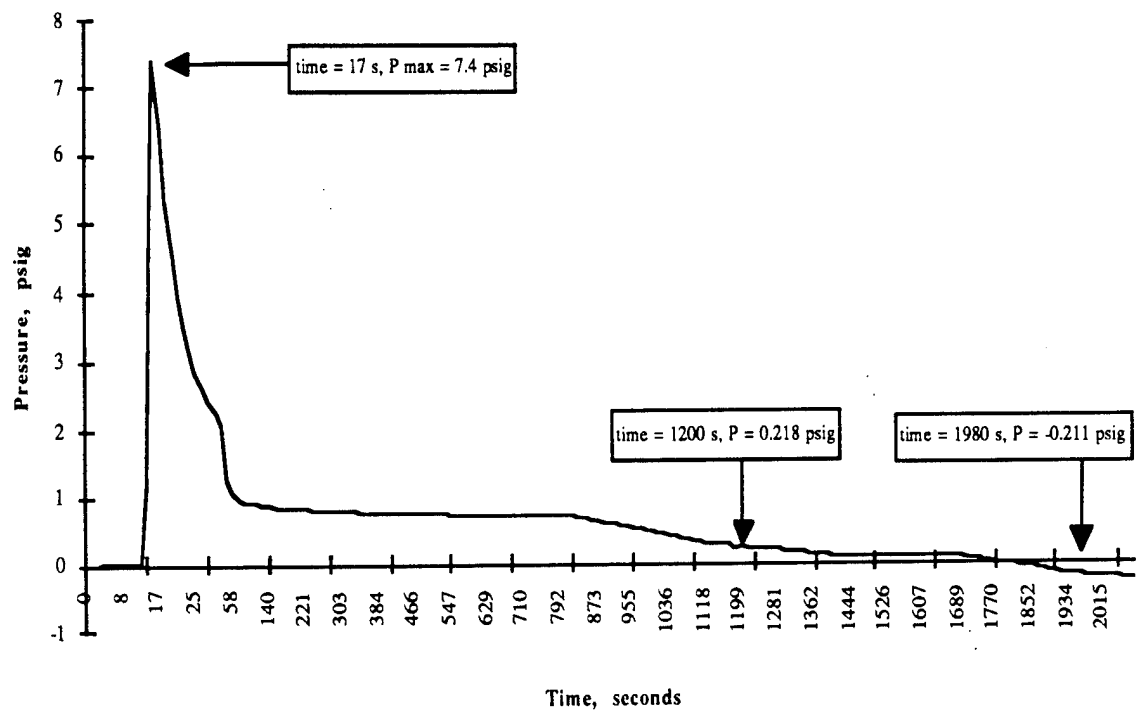


Figure 11. Chamber Pressure, Test 2, A-50/N₂O₄ Oxidizer-Rich Combustion.

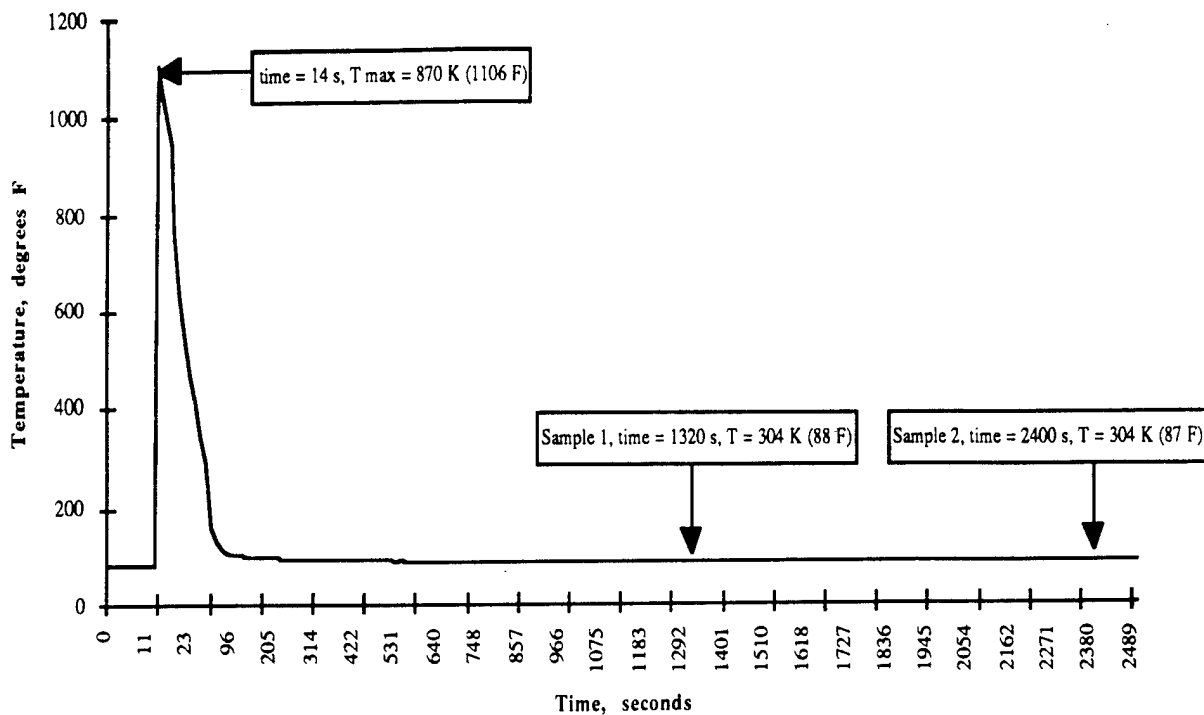


Figure 12. Chamber Gas Temperature, Test 3, A-50/N₂O₄ Stoichiometric Combustion.

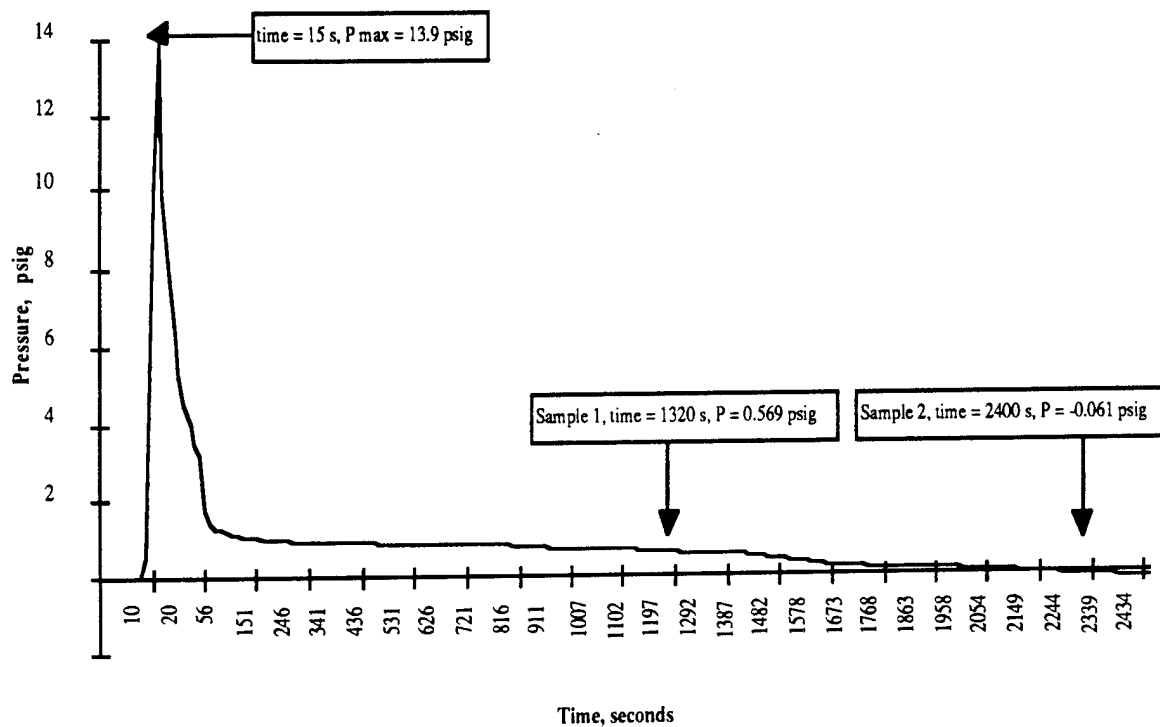


Figure 13. Chamber Pressure, Test 3, A-50/N₂O₄ Stoichiometric Combustion.

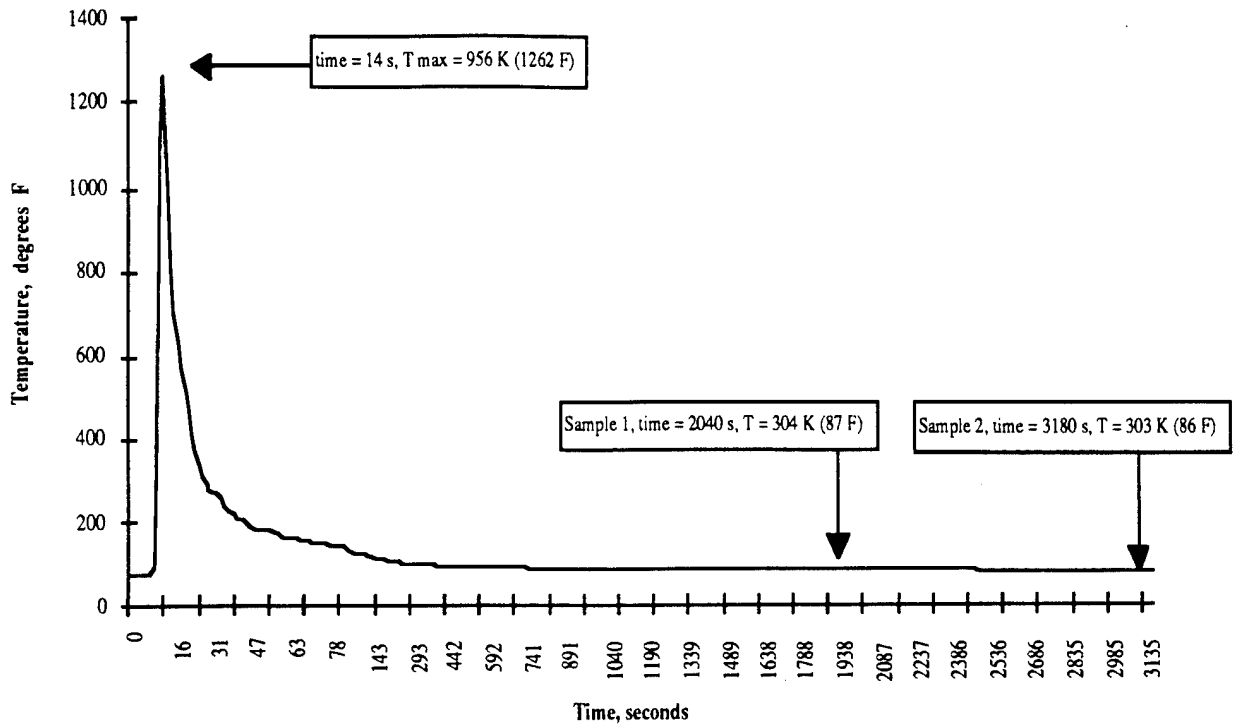


Figure 14. Chamber Gas Temperature, Test 4, A-50/N₂O₄/PBAN Solid Propellant.

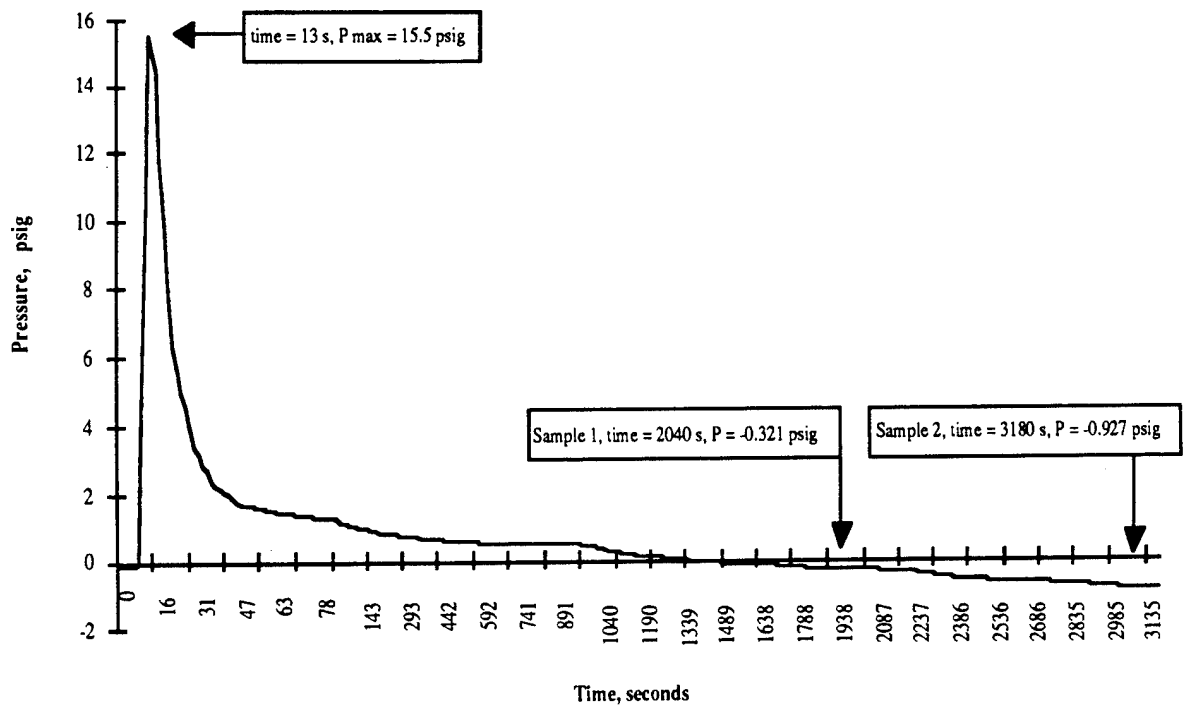


Figure 15. Chamber Pressure, Test 4, A-50/N₂O₄/PBAN Solid Propellant.

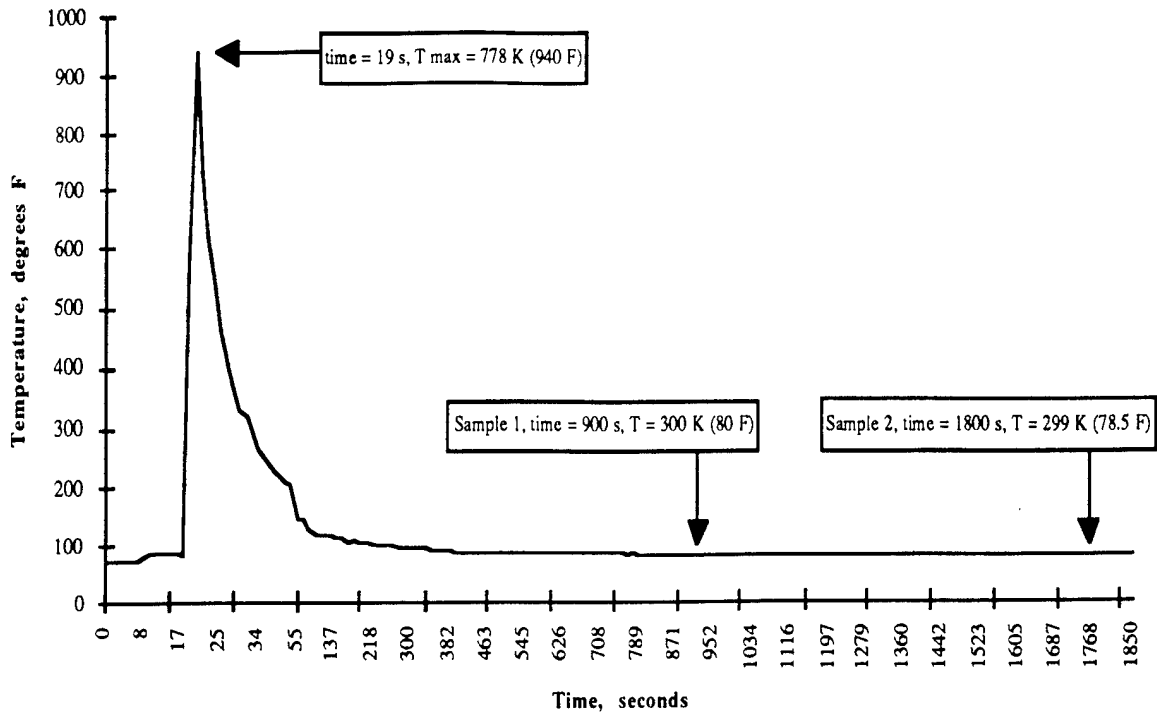


Figure 16. Chamber Gas Temperature, Test 5, RP-1/GOX Fuel-Rich Combustion.

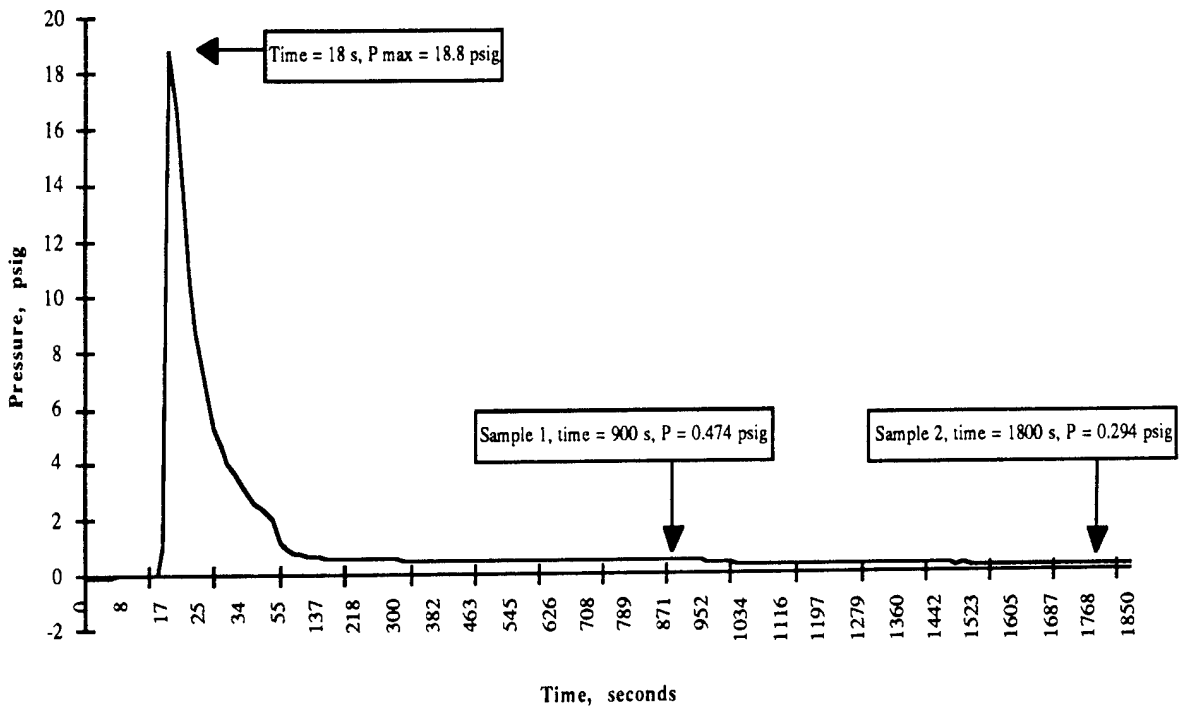


Figure 17. Chamber Pressure, Test 5, RP-1/GOX, Fuel-Rich Combustion.

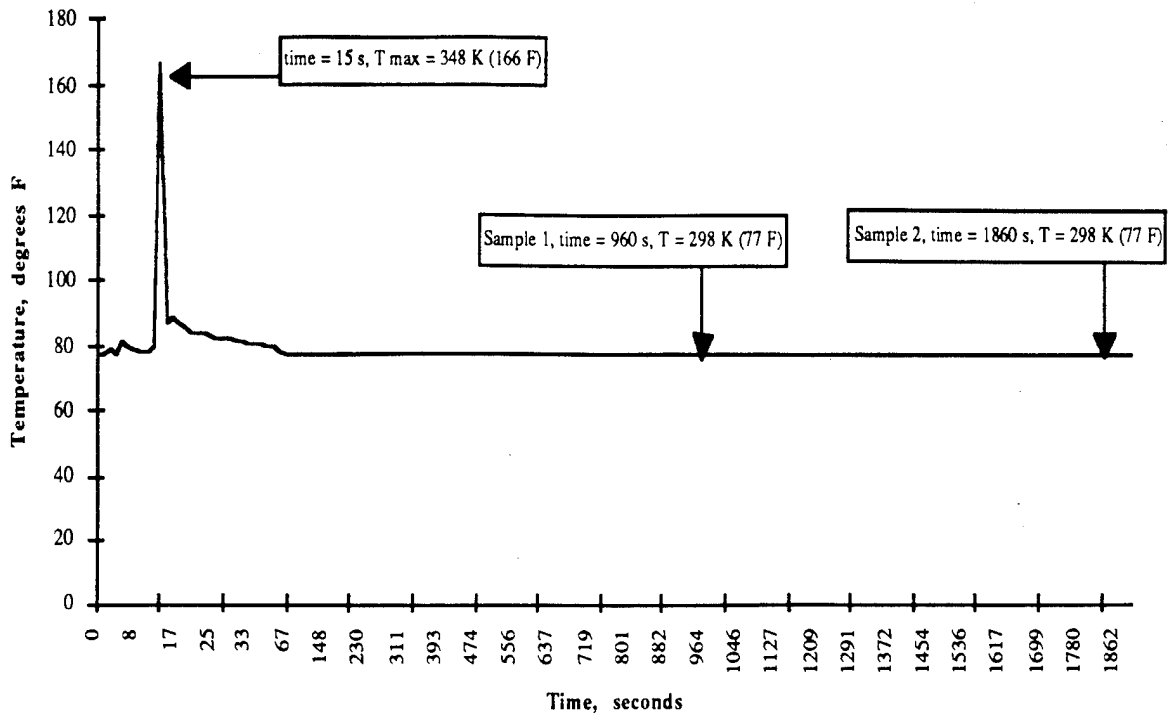


Figure 18. Chamber Gas Temperature, Test 6, RP-1/GOX Oxidizer-Rich Combustion.

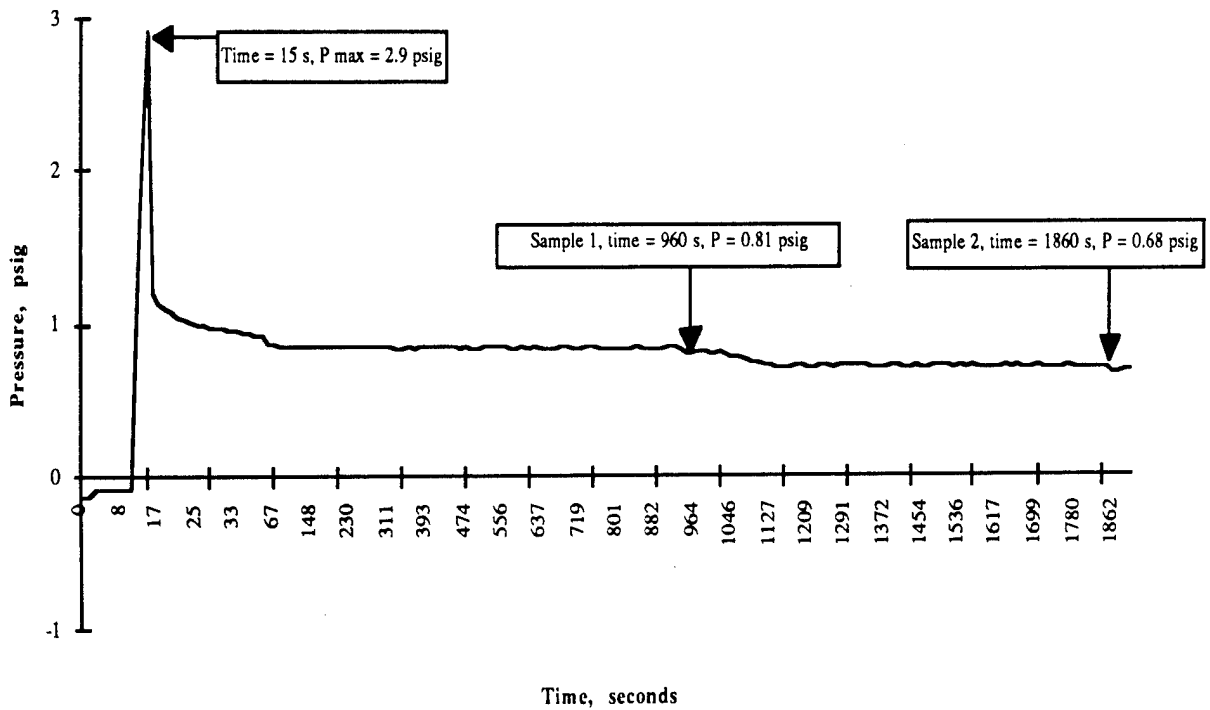


Figure 19. Chamber Pressure, Test 6, RP-1/GOX Oxidizer-Rich Combustion.

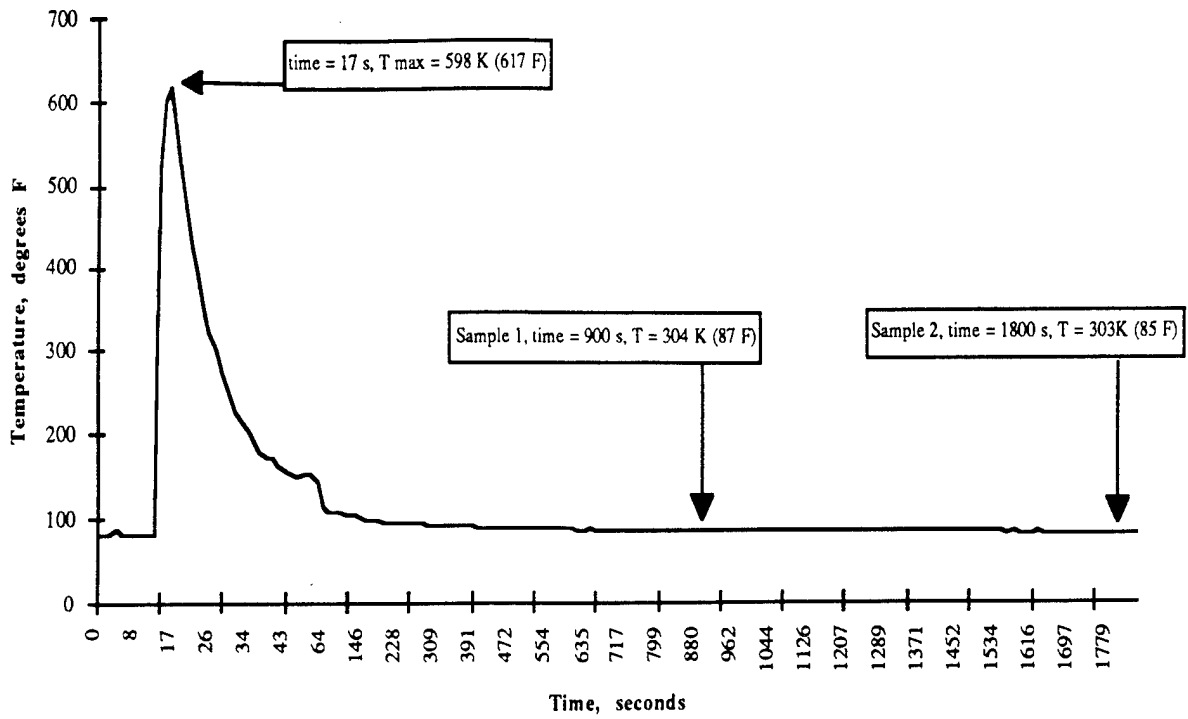


Figure 20. Chamber Gas Temperature, Test 7, RP-1/GOX Stoichiometric Combustion.

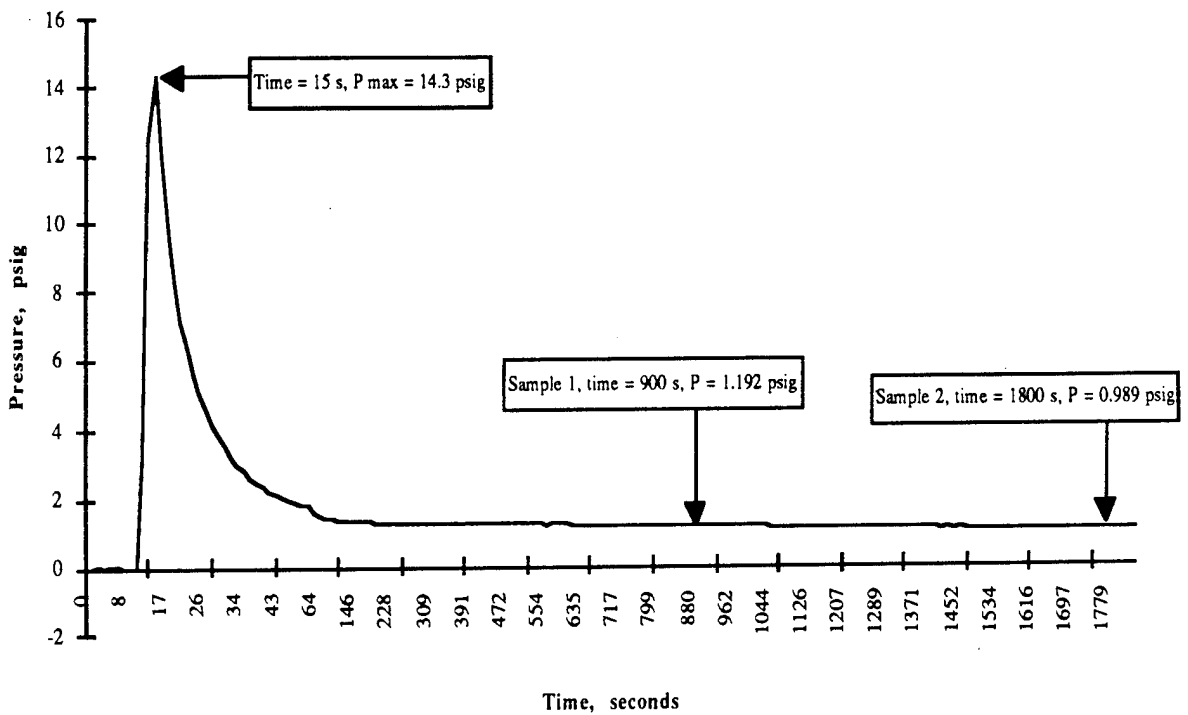


Figure 21. Chamber Pressure, Test 7, RP-1/GOX Stoichiometric Combustion.

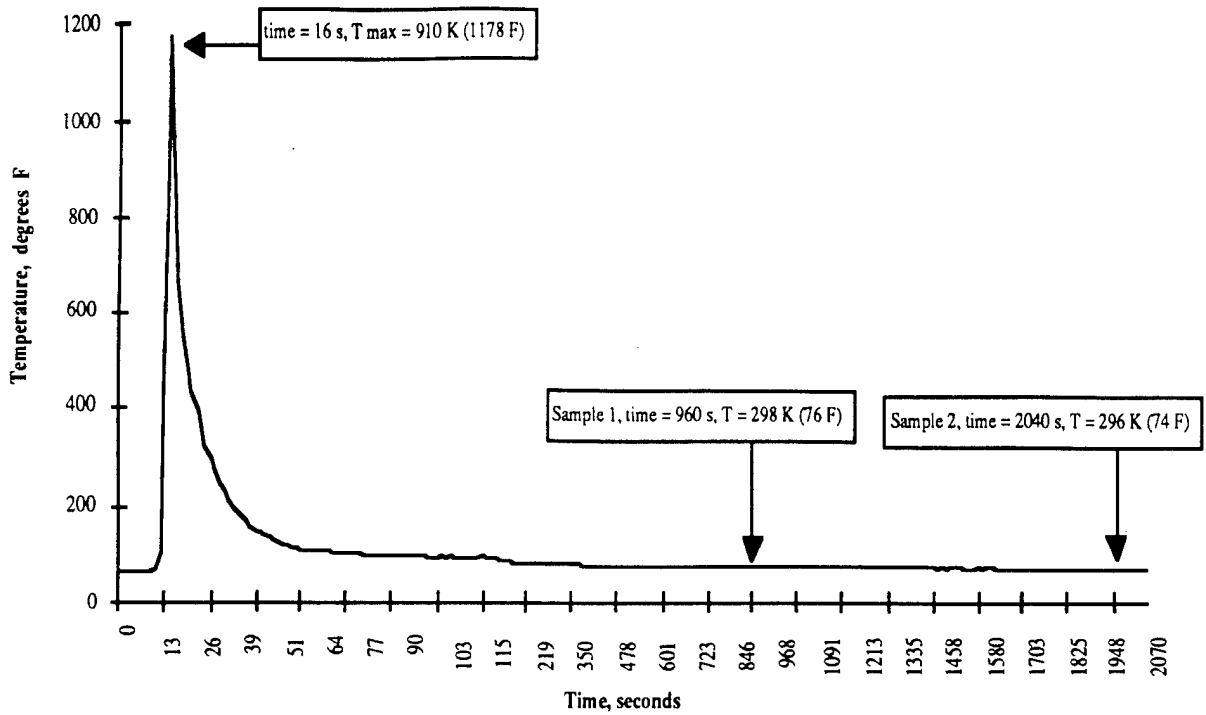


Figure 22. Chamber Gas Temperature, Test 8, A-50/N₂O₄, RP-1/GOX, Castor IVA.

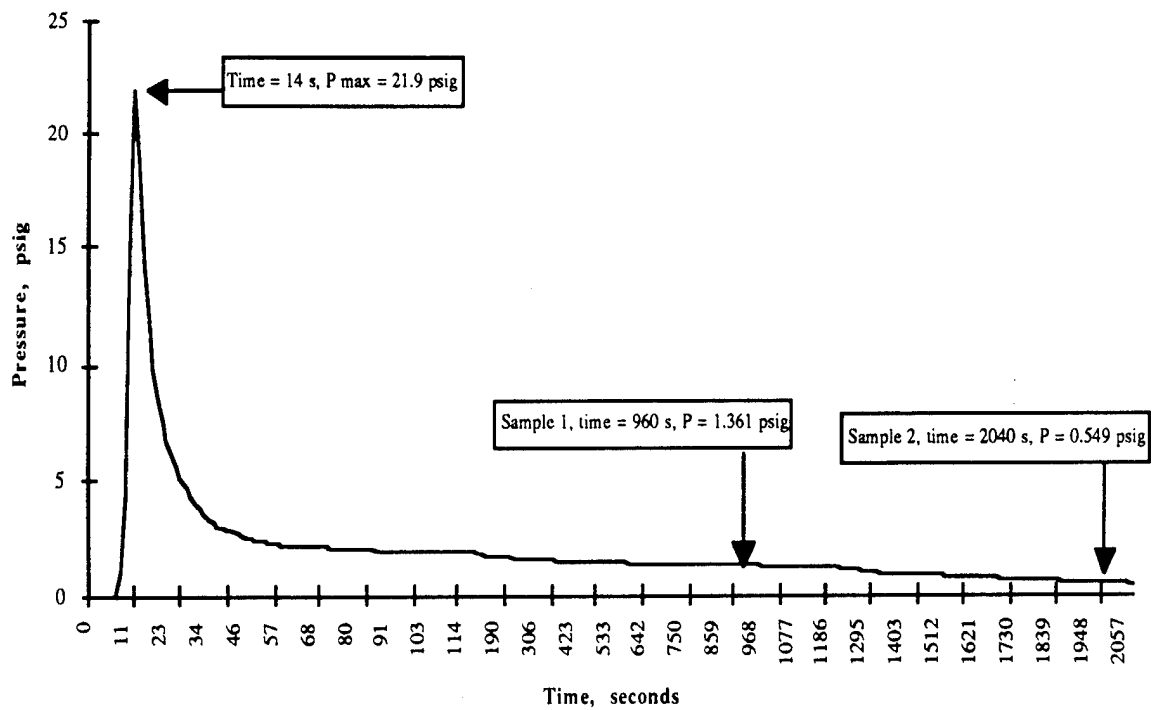


Figure 23. Chamber Pressure, Test 8, A-50/N₂O₄, RP-1/GOX, Castor IVA.

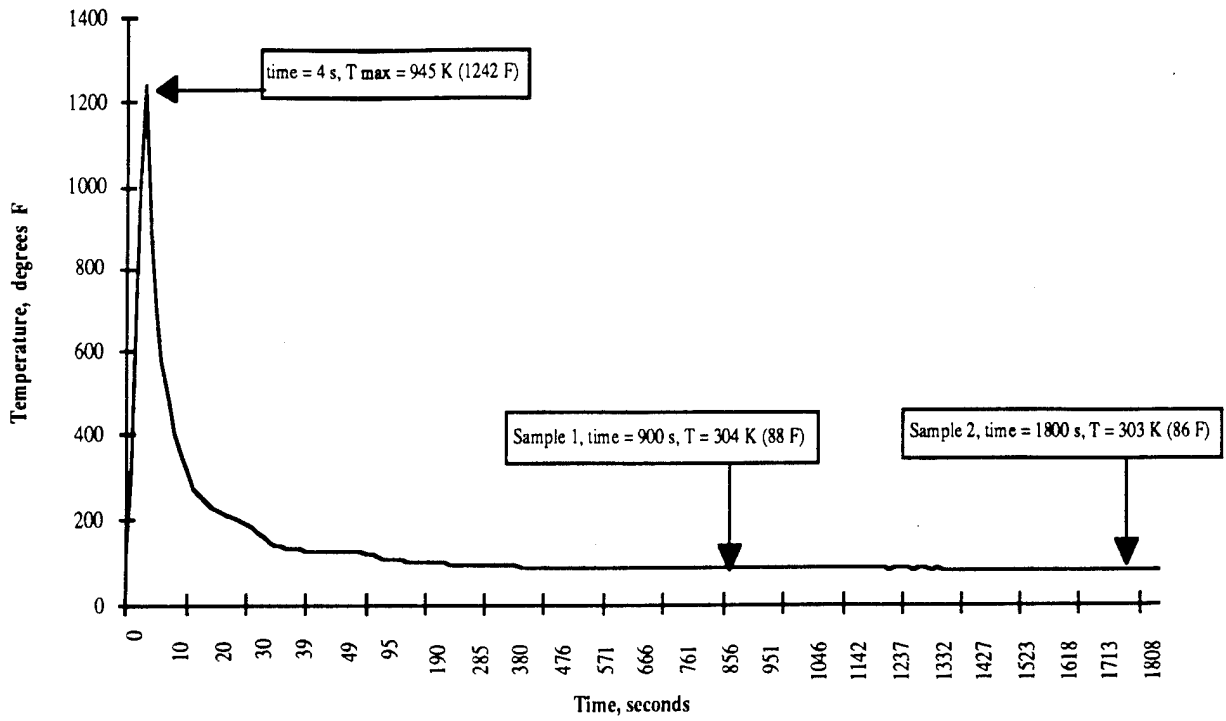


Figure 24. Chamber Gas Temperature, Test 9, PBAN.

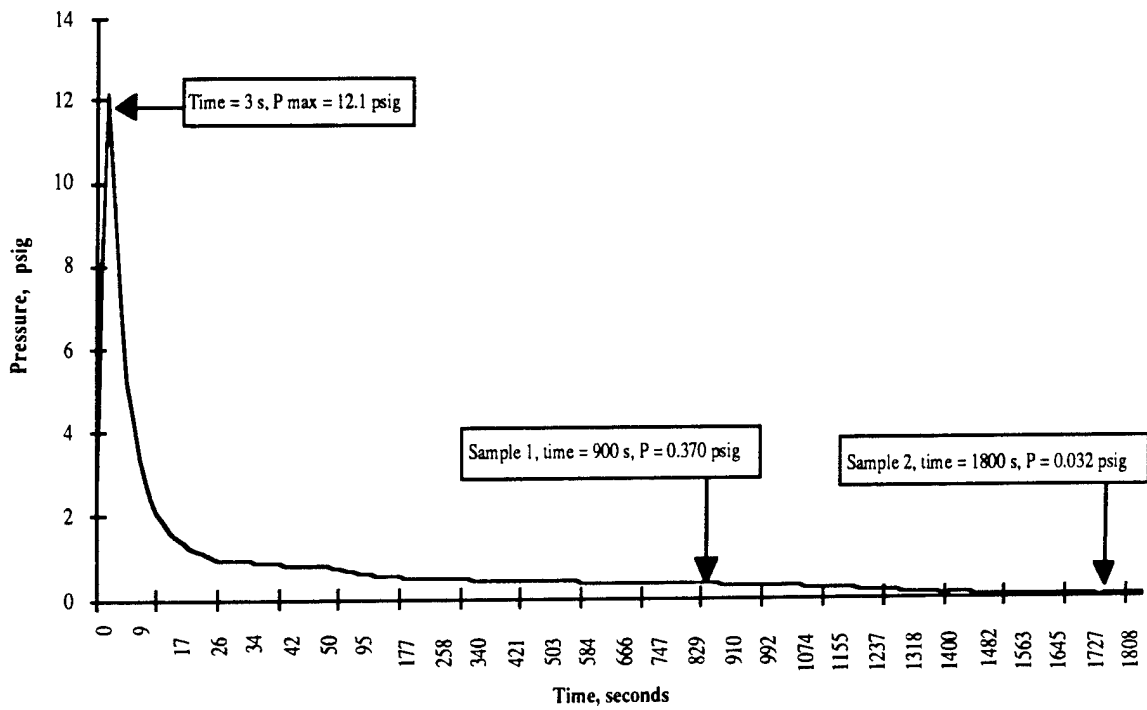


Figure 25. Chamber Pressure, Test 9, PBAN.

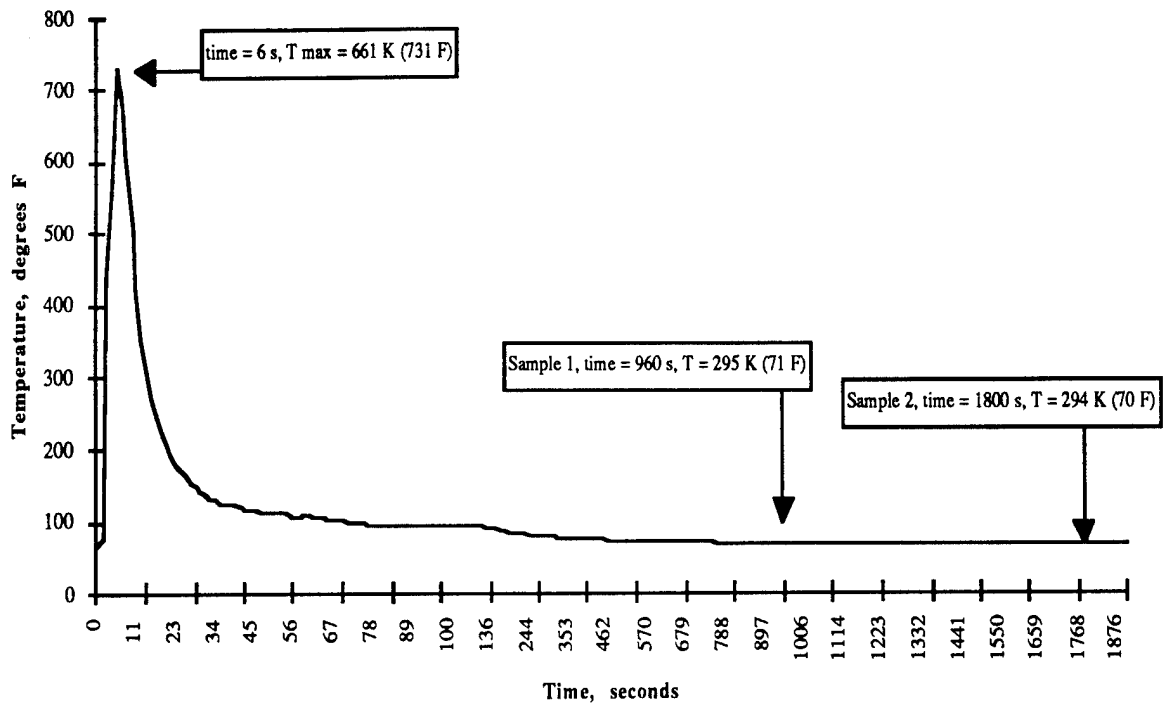


Figure 26. Chamber Gas Temperature, Test 10, Castor IVA.

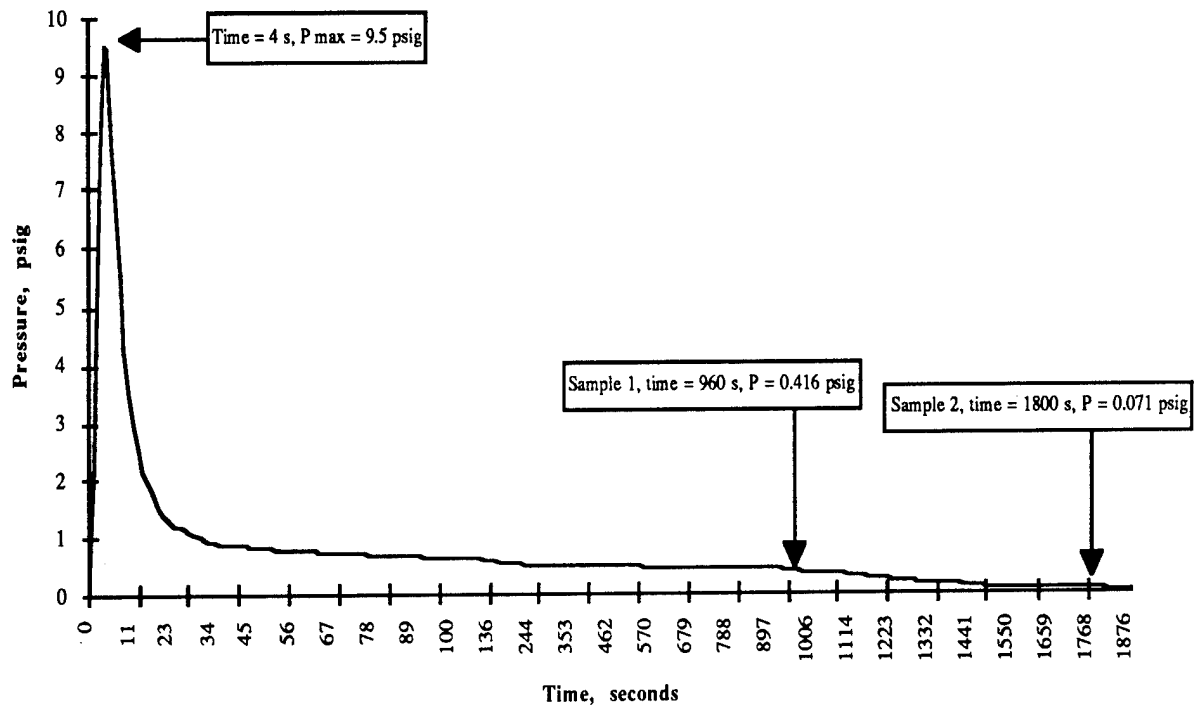


Figure 27. Chamber Pressure, Test 10, Castor IVA.

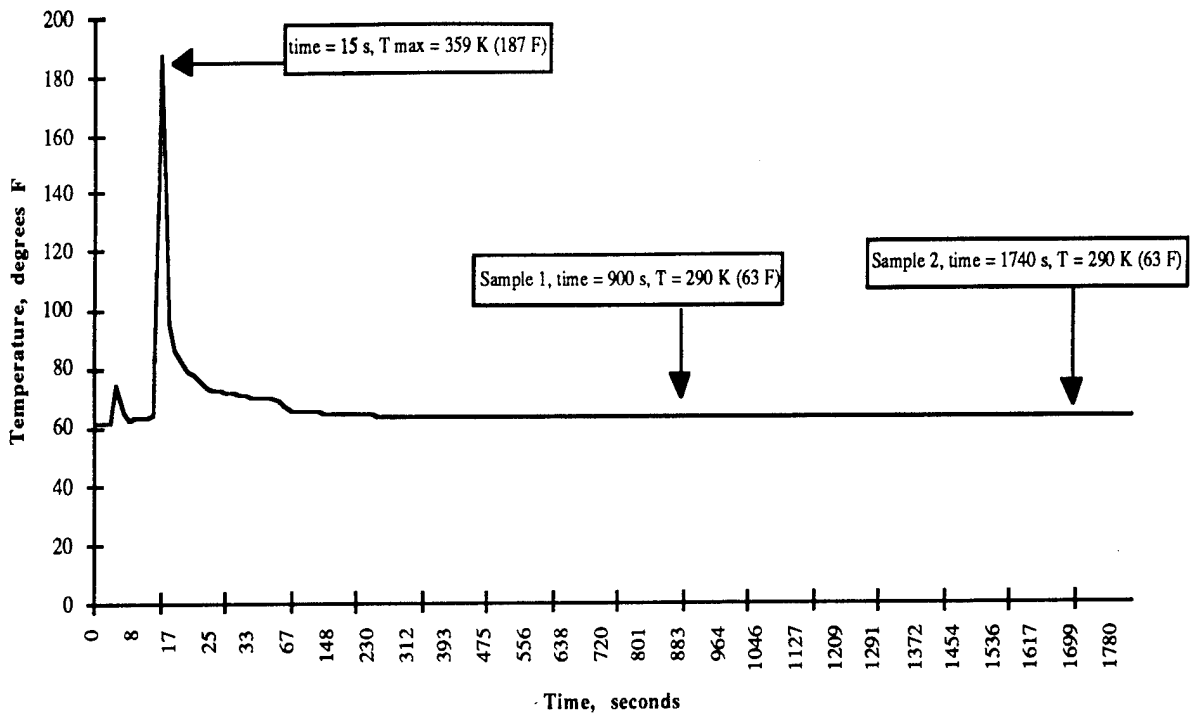


Figure 28. Chamber Gas Temperature, Test 11, RP-1/GOX Oxidizer-Rich Combustion (Repeat).

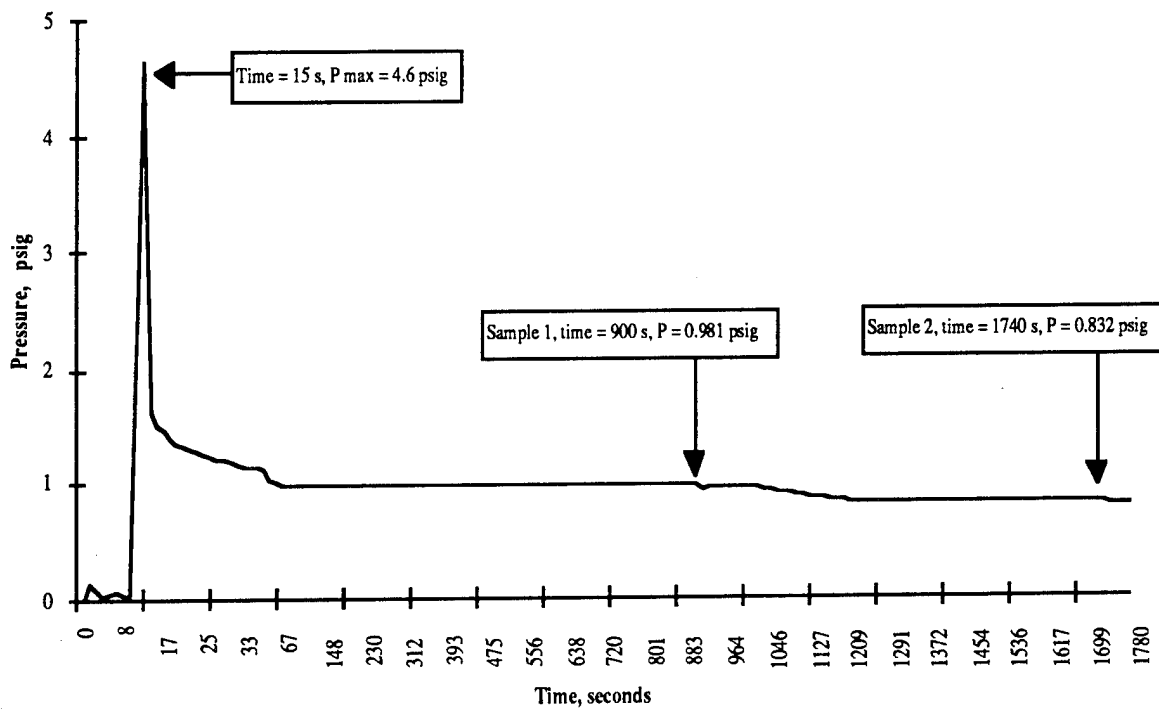


Figure 29. Chamber Pressure, Test 11, RP-1/GOX Oxidizer-Rich Combustion (Repeat).

SECTION IV

DISCUSSION

A. OPEN CHAMBER COMBUSTION TESTS

The series of open chamber combustion tests, observed visually and on high speed video photography, proved invaluable in the design and optimization of future tests conducted with the chamber dome sealed. In particular, the degree of combustion, and thus the quality of the test, was heavily dependent on a variety of operating parameters. The ability to align the nozzles to generate a reproducible and efficient impingement pattern proved difficult. The nozzle used for the delivery of Aerozine-50 was especially troublesome, and repeated clogging of this nozzle occurred. To mitigate and improve combustion concerns, nozzles were cleaned with isopropyl alcohol and water before each use, and the impingement pattern was aligned using water as a test fluid. Small filters were also placed upstream of each solenoid valve to trap any particulates in the delivery system. In some cases, a shim was added as the nozzle was tightened to the chamber, which improved the alignment and impingement of the opposing streams. A small machining burr was observed in the 0.018-inch diameter nozzle used for dinitrogen tetroxide. Unfortunately, this burr was not noticed until most of the testing (including the sealed chamber testing) was completed. This burr could account for some of the incomplete combustion of the A-50 with N_2O_4 , as its effect was to jitter the N_2O_4 stream in and out of the intersection path with the fuel.

1. RP-1/GOX Tests

Good combustion was obtained between RP-1 and gaseous oxygen when the flame was observed through the chamber viewport. A minor sputtering effect (unstable combustion) occurred during the early stages of the burn, and was attributed to ejection of the dead volume of the oxygen line (which contained room air). To alleviate this concern, the dead volume of the oxygen line was purged with pure oxygen prior to combustion. Likewise, dead volumes in the RP-1 delivery system (up to the nozzle exit) were charged with fresh RP-1 before initiation of flow and subsequent combustion. To further optimize the burning process, the ignitor circuit was carefully aligned at the intersection point of the fuel and oxidant streams prior to each test.

2. A-50/ N_2O_4 Tests

Combustion tests with Aerozine-50 and dinitrogen tetroxide proved challenging. In addition to problems previously described (nozzle alignment problems and clogging of orifices), excess dinitrogen tetroxide was observed at the completion of the reaction. This condition persisted after nozzles were carefully cleaned, inspected, and aligned prior to each test. The parameters that produced the most efficient combustion, with the highest evolution of heat and lowest amount of residual dinitrogen tetroxide, were the delivery pressures of opposing fuel and oxidant streams. Initial tests designed for stoichiometric combustion of Aerozine-50 with dinitrogen tetroxide incorporated matched diameter nozzles (0.0128 inches) for the two streams, ejected at a delivery pressure of 148 psig for N_2O_4 and 47 psig for Aerozine-50. Although the delivery pressures resulted in stoichiometric delivery of the propellants (O/F mole ratio = 1.0), the resultant burn was extremely poor, with a low heat content and a large quantity of residual dinitrogen tetroxide released. Residual Aerozine-50 was also likely released into the chamber, but this chemical was not visually evident, as was the excess dinitrogen tetroxide which was dark red in color. When the delivery pressures of the two streams were matched (keeping the orifice diameters constant at 0.0128 inches), a substantial improvement in the burning process was observed. In this case, the combustion flame was white hot, and only a minor amount of residual dinitrogen tetroxide was observed. These results were obtained for the matched flow condition, even though the mixture ratio was fuel-rich under these conditions (O/F = 0.56). All future tests

involving Aerozine-50 and dinitrogen tetroxide were therefore designed such that the delivery pressures would be matched to the greatest extent possible, and the mixture ratio varied as needed by changing nozzle diameters.

3. Solid Propellant Tests

Tests with the solid propellants (PBAN and Castor IVA) were performed outdoors, as well as inside the combustion chamber, to demonstrate ignition of these propellants at ambient pressure. There was initially some concern that ignition would be difficult because these propellants are designed to combust at high pressures typical in a rocket exhaust motor (1000 pounds per square inch). However, passing an electrical current from a 28-volt power supply through a high resistivity nichrome wire ignited these propellants efficiently and reproducibly. The time required for the complete combustion of a ten gram sample of propellant, using a single strand of wire embedded into the propellant, was between 7 and 10 seconds at ambient temperature and pressure. The only problem was the "dancing" of the propellant block once ignition had begun. To remedy this problem, a small stainless steel wire cage was fabricated to restrict the movement of the propellant block during combustion. It was also noted that when the ignitor wire was placed on the top surface of the propellant block, the combustion products of the solid propellant were ejected upwards into the chamber and seriously disrupted the combustion flow of the liquid propellants. The block was therefore rotated 90 degrees to eject the combustion gases laterally inside the chamber and to minimize interference with the combustion of the liquid propellants.

B. CHAMBER RECOVERY TESTS

Recovery of chemicals introduced into the chamber was very good for carbon monoxide, carbon dioxide, and dinitrogen tetroxide. Recovery of Aerozine-50, an equal weight mixture of hydrazine and unsymmetrical dimethylhydrazine was inconsistent, and for hydrazine was less than desired. At prevailing chamber temperatures (nominally 25°C during sampling), excess N_2H_4 and UDMH were expected to be completely vaporized, collected, and analyzed. Another removal mechanism for hydrazine, such as surface interactions with the chamber wall, reaction with residual water to form condensed hydrazine hydrate, liquid-vapor heat transfer problems (an extended time may have been required for hydrazine to vaporize under these conditions), or inhomogeneity of the chamber gas due to poor mixing, may have accounted for these poor recoveries.

C. PROPELLANT INTERACTION TESTS

A brief discussion of the results of each propellant interaction test is presented below.

1. Test 1, A-50/ N_2O_4 Fuel-Rich Combustion

Several important observations were made during the course of conducting this test and analyzing the chemical data. Expected thermal decomposition products of Aerozine-50 (ammonia, hydrogen, and methane) were not formed in appreciable amounts. These materials, if formed, were combusted with residual air to form expected combustion products (nitrogen, water vapor, and carbon dioxide). Carbon monoxide was converted to carbon dioxide, which was expected thermodynamically upon cooling of the combustion gases to room temperature. Residual oxygen concentrations approximated the predicted values, but these values were based on the combustion of excess Aerozine-50 with air, which may not have occurred. No residual hydrazine or 1,1-dimethylhydrazine was observed in the vapor phase. A total carbon balance indicated that of the initial amount of 1,1-dimethylhydrazine, 60 percent was reacted to form carbon dioxide, and the remaining was unaccounted for. Intermediate combustion products, condensed hydrazine hydrates, other condensation products, and reactions with carbon dioxide to form carbazic acid derivatives are believed to be the major removal mechanisms for Aerozine-50.

Nitrosodimethylamine, an established animal carcinogen, was observed both in the gas phase and in the condensed phase. Temperature and pressure data indicated less combustion than predicted.

2. Test 2, A-50/N₂O₄ Oxidizer-Rich Combustion

In this test, hydrogen and carbon monoxide were absent, again suggesting afterburning with residual air. The oxygen concentration analyzed was depressed from expected values. Residual nitrogen dioxide was 47 percent higher than predicted values, suggesting poor mixing or poor combustion of the impinging streams, as had been noted for this propellant combination. Nitrosodimethylamine was also observed in the gas phase, although it was not predicted for this test condition. Several hydrazine and 1,1-dimethylhydrazine oxidation products were observed in the condensed phase, as were inorganic nitrates (or nitric acid). These results indicated that unreacted dinitrogen tetroxide remained after the combustion, and that intermediate combustion products of both Aerozine-50 and dinitrogen tetroxide were formed and condensed upon cooling of the chamber gas.

3. Test 3, A-50/N₂O₄ Stoichiometric Combustion

The results of this test were clearly not representative of an efficient stoichiometric propellant combustion. After combustion and cooling, the chamber gas consisted of 1.7 percent nitrogen dioxide (NO₂), which was 44 percent of the original quantity of dinitrogen tetroxide. Although no excess hydrazine or 1,1-dimethylhydrazine was observed in the vapor phase, the gas pressure was significantly lower than expected, again suggesting that incomplete combustion or adsorption of the fuels on the chamber walls had occurred. Excess dinitrogen tetroxide may have resulted from nozzle instability or from the difficulty in properly combusting these two propellants.

4. Test 4, A-50/N₂O₄, PBAN Solids

The purpose of this test was to demonstrate the interactions of the Titan IV liquid and solid propellants. This test had the most unusual and unpredicted result. After completing the test and opening the chamber, a large quantity of water soluble, bronze-colored condensate had coated the entire upper hemisphere of the chamber (see photograph in Figure 30). A Fourier Transform Infrared (FTIR) analysis of this residue indicated that the material was an inorganic nitrate with similar absorption frequencies as ammonium nitrate (Figure 31). Because of the large amount of the deposit, a further investigation of the identity of this material is warranted. Other observations for this test were an efficient conversion to carbon dioxide (86 percent of available carbon from the A-50 and PBAN were converted to carbon dioxide), formation of nitrosodimethylamine (as well as hydrazine oxidation products) in the gas and condensed phases, formation of chlorinated hydrocarbons in the gas phase, and the appearance of extremely high nitrate values on the witness plate. These values also suggested the condensation of an inorganic nitrate. In addition, residual nitrogen dioxide, although evident, was less prominent than in the previous test. These results suggest that the formation of condensation products, presumably between nitrogen dioxide and the solid propellant combustion products, is a major fate for these propellants.

5. Test 5, RP-1/GOX Fuel-Rich Combustion

Results of this test indicated very efficient combustion, including combustion of excess RP-1 fuel. Carbon balance showed that 84 percent of all RP-1 was combusted. Hydrogen and carbon monoxide were not detected, indicating afterburning of these materials to form water vapor and carbon dioxide had occurred. Oxygen levels were depressed from expected values, which was consistent with the increased combustion and afterburning of hydrogen, carbon monoxide, and residual RP-1. Some residual organics were observed in the vapor phase. These were attributed to the small amounts of residual RP-1 that did not combust with gaseous oxygen or experience afterburning with air.

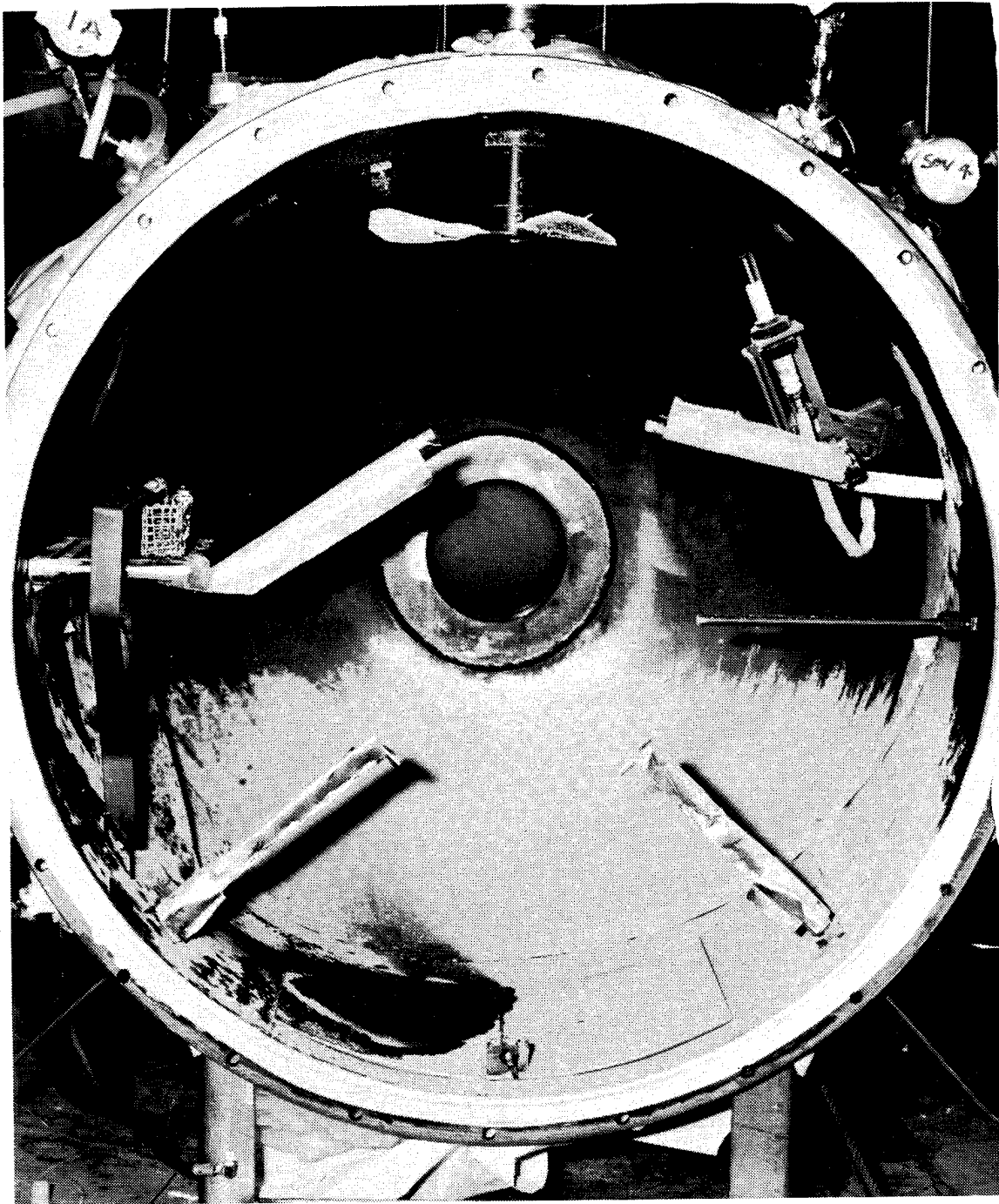


Figure 30. Chamber Residue after Completion of Test 4.

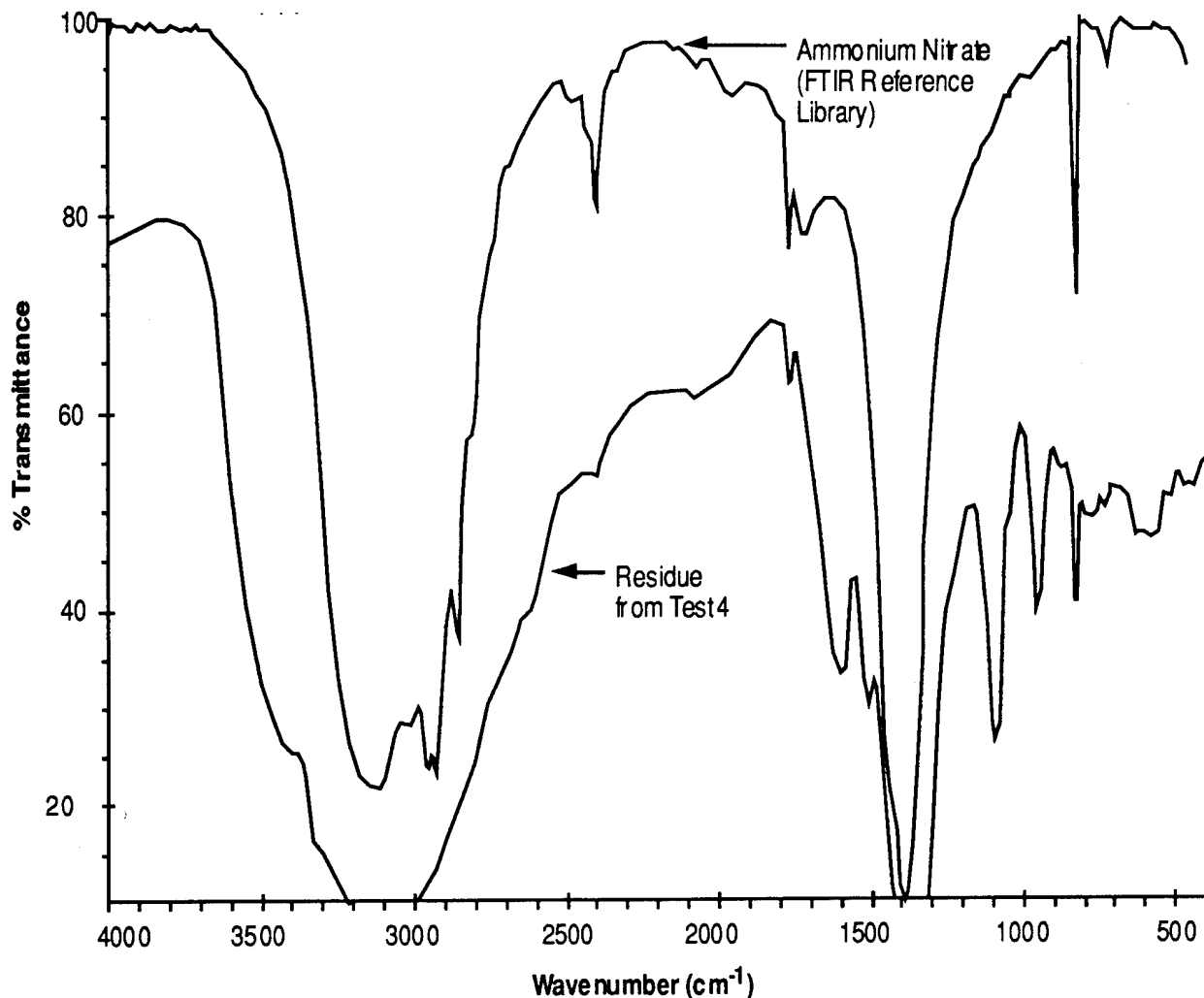


Figure 31. FTIR Spectrum, Residue from Test 4.

6. Test 6, RP-1/GOX Oxidizer-Rich Combustion

Results of this test indicated very poor combustion of the RP-1 fuel with oxygen. Poor combustion was also noted during visual inspection of the combustion process through the chamber view port. The mixture seemed to spark or flare slightly at the completion of the 2 second burn period. Less than 5 percent of the available RP-1 combusted to form carbon dioxide based on a carbon balance. In addition, the oxygen level analyzed was considerably higher than expected values, and pressure and temperature rises were significantly lower than expected. The poor combustion may have been due to the high oxygen delivery pressures required for the performance of this test (520 pounds per square inch). This delivery pressure may have resulted in an unstable combustion flame. The poor combustion may also have occurred from the fuel-lean combustion condition. Because of the exceptionally poor combustion, this test was repeated. Results of this repeated test (Test 11) were identical to the results for Test 6.

7. Test No. 7, RP-1/GOX Stoichiometric Combustion

The results of this test indicated that approximately 60 percent of the initial quantity of RP-1 was combusted to form carbon dioxide. Pressure and temperature rises were about 50 percent lower than expected. Oxygen levels were higher than predicted amounts, also indicating that incomplete combustion had occurred.

8. Test No. 8, A-50/N₂O₄, RP-1/GOX, Castor IVA

This test was designed to simulate the interactions of the liquid and solid rocket propellants contained on the Delta II launch vehicle. Results of this test indicated that conversion of available carbon to carbon dioxide was only 33 percent efficient. Pressure and temperature rises were significantly lower than expected, also indicating incomplete and poor combustion. Excess nitrogen dioxide was observed, but was low (0.228 volume percent NO₂), corresponding to 6 percent of the initial amount of dinitrogen tetroxide. Hydrogen chloride gas was not observed in appreciable amounts. Hydrazine and 1,1-dimethylhydrazine oxidation products (including nitrosodimethylamine), hydrocarbon residues, and nitrated hydrocarbons were observed in the condensed phase. These results indicated poor combustion of the liquid propellants, and suggested that excess dinitrogen tetroxide had reacted with RP-1 and/or products of the solid propellant to form unique and complex combustion products. In addition, the disruption of the flow dynamics of the propellants may have contributed to the inefficient combustion.

9. Test 9, PBAN

This test resulted in very good combustion, with results generally in agreement with predicted values. Hydrogen, carbon monoxide, and methane were not detected and were assumed to be combusted in afterburning with air. Oxygen levels were slightly lower than predicted values, and were consistent with afterburning processes. Conversion of available carbon to carbon dioxide (carbon balance) was 87 percent. Hydrogen chloride was not detected in the gas phase, but may have condensed from the chamber as a hydrated acid. The pressure rise was slightly lower than predicted values, and the temperature rise was higher than predicted values. The latter result may have been due to the geometric placement of the solid in the chamber.

10. Test 10, Castor IVA

The combustion of the Castor IVA propellant was not as efficient as the PBAN combustion (pressure rise was significantly lower than expected), but this material generally displayed good conversion to its theoretical combustion products. Conversion of available carbon to carbon dioxide (carbon balance) was 80 percent. Hydrogen, carbon monoxide, and methane were not detected above background levels. Oxygen levels were slightly lower than predicted values. Hydrogen chloride was detected in the vapor phase, but was lower than its expected value, again suggesting the condensation of the hydrated acid.

SECTION V

CONCLUSIONS

Based on the results of these tests, the following conclusions with respect to the combustion of liquid and solid rocket propellants in air at prevailing temperature and pressure conditions are presented:

A. Combustion tests using Aerozine-50/dinitrogen tetroxide, RP-1/GOX, and solid rocket propellants were successfully conducted and yielded chemical composition data essential to the prediction of toxic gases released from a launch vehicle accident. High quality test results were obtained, and the primary objectives of this program were achieved.

B. Excess Aerozine-50 did not appear as vaporized propellant. This chemical is likely to be removed from the combustion gases as a variety of complex condensates, including hydrazine hydrates, intermediate combustion products (such as formaldehyde dimethylhydrazine and hydrazine azine), nitrosodimethylamine (an established carcinogen), and perhaps hydrazine nitrate residues. The identification of the major components of these condensed residues is important because the carbon balance accounts for only 60 percent of the original UDMH fuel, and because these products are at least as toxic as the parent material.

C. Residual nitrogen dioxide was observed in all tests involving dinitrogen tetroxide propellant. The appearance and quantification of this material indicate that excess nitrogen dioxide released from accidents likely remains as pure vapor, except in its interaction with solid rocket propellants and unreacted RP-1.

D. Reactions of RP-1 with gaseous oxygen produced varied results, ranging from near complete combustion (fuel-rich case), to intermediate combustion (stoichiometric case), to extremely poor combustion (oxidant-rich case). Predictably, vaporized and condensed fractions of the unreacted hydrocarbon fuel were observed in tests in which incomplete combustion had occurred.

E. Solid propellants exhibited very complete combustion and produced results that agreed well with predicted values. Hydrogen chloride was absent in the vapor phase and this was attributed to the interaction with water vapor and subsequent condensation as the hydrated acid.

F. Reactions involving the combustion of liquid and solid rocket propellants produced results in poor agreement with predicted values, and the formation of condensates was significant. Water soluble nitrates appeared to be the primary condensed products. A more detailed investigation of these products is warranted.

G. Significant afterburning of residual hydrogen and carbon monoxide to form water vapor and carbon dioxide had occurred in every test.

H. Thermal decomposition products of the hydrazines (ammonia, hydrogen, and methane) were absent, indicating that if formed, these materials also experienced significant afterburning with air.

I. Combustion reactions observed in these tests may differ in a launch vehicle accident. In such an accident, an unconfined vapor cloud is formed, the extent of mixing is influenced by prevailing wind conditions, and scaling considerations for heat and mass transport are important. Results of this study, however, are a significant improvement over theoretical predictions of the release of toxic launch gases.

SECTION VI

RECOMMENDATIONS

Areas requiring further investigation are presented in the following paragraphs. These additional studies are proposed to improve the prediction of toxic chemicals arising from a launch vehicle accident. They are listed in order of priority.

A. FATE OF UNREACTED AEROZINE-50

Aerozine-50/dinitrogen tetroxide tests were instrumental in showing the absence of fuel vapors released after mixing and combusting these propellants. This unexpected result may have beneficial implications for toxic chemical dispersion modeling, although chamber wall interactions may also have contributed to this observation. The fate of unreacted fuels in an accident environment remains uncertain and should be evaluated further to better model these materials for launch safety operations. The discovery that residual hydrazine and unsymmetrical dimethylhydrazine were absent in the vapor phase after combustion, and that only 60 percent of the unsymmetrical dimethylhydrazine was converted to carbon dioxide, indicate that a significant removal mechanism for excess Aerozine-50 is by reaction or by condensation. A mass balance of reacting Aerozine-50 should be performed and quantification of released chemicals should be determined. These chemicals may include oxidation products (carbon dioxide, nitrogen, and water vapor), incomplete oxidation products (nitrosodimethylamine, formaldehyde dimethylhydrazone, and other complex chemicals), and condensed Aerozine-50 residues.

B. NITROSODIMETHYLAMINE EVALUATION

Identification of nitrosodimethylamine (NDMA) in gas and condensed samples confirmed the expected presence of this chemical in the Aerozine-50/dinitrogen tetroxide reactions. The contribution of this chemical to the total chemical inventory in a fireball cloud is believed to be small.⁹ Because this chemical is an established carcinogen, and because previous studies did not include interactions with dinitrogen tetroxide or other rocket propellants, quantification of NDMA formed in propellant combustion reactions would improve existing modeling efforts.

C. IDENTIFICATION OF CONDENSED RESIDUES FROM A-50/N₂O₄/PBAN TESTS

The deposition of a bronze-colored residue from the A-50/N₂O₄/PBAN test was a significant finding. This material was predominant in the chamber and is believed to be a significant removal mechanism for excess dinitrogen tetroxide under these conditions. The implication for launch vehicle abort modeling is the deposition of condensate near the accident site. Identification of this residue would refine the existing model and help establish this chemical as a toxic or non-toxic material.

⁹A study performed by Martin Marietta in 1983 suggests that only 0.002% of available UDMH is air oxidized to NDMA. Prince, S. Atmospheric Dispersion of Hypergolic Liquid Rocket Fuels. Task 1 Plume Atmosphere Interaction May 18, 1993. Martin Marietta Corporation.

D. AEROZINE-50 RECOVERY EVALUATION

Recovery tests performed with Aerozine-50 demonstrated that excess fuel introduced into the chamber would be detected upon chemical analysis. This result is important because residual Aerozine-50 was absent in all the combustion tests. Chemical recoveries for hydrazine and unsymmetrical dimethylhydrazine were inconsistent and the poor recoveries may have been due to chamber wall interactions or incomplete vaporization. These results should be investigated further to better understand the vaporization process of Aerozine-50 in the metal chamber, and to apply these results more consistently to combustion tests.

E. IMPROVE NOZZLE PERFORMANCE

Combustion tests between Aerozine-50 and dinitrogen tetroxide gave good results, and demonstrated the difficulty in ensuring complete combustion of these hypergolic propellants. In a launch vehicle accident, incomplete mixing is expected to occur and release of vaporized dinitrogen tetroxide is expected. This prediction is supported by the test results, in which nitrogen dioxide was observed in every test involving these propellants. Instabilities in nozzle performance may have contributed to incomplete combustion of Aerozine-50 and dinitrogen tetroxide. Improvements in nozzle design and/or operating parameters of these nozzles would result in more efficient combustion, and improvements in the prediction of toxic gases resulting from accidents involving these propellants. In particular, the release and quantification of residual dinitrogen tetroxide and Aerozine-50 under non-stoichiometric combustion conditions can be greatly improved by correcting these instability problems.

F. FATE OF HYDROGEN CHLORIDE

Because hydrogen chloride gas is predicted from the combustion of solid rocket propellants, and because no hydrogen chloride was discovered in the vapor phase, the fate of this material is also important to launch vehicle abort monitoring. A mass balance of released HCl, including vapor-phase HCl, condensed (aqueous) phase HCl, and any reaction products containing chlorine should be determined.

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APPENDIX A
TEST DATA SHEETS

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TABLE A-1. TEST OA DATA SHEET.

Date 4-16-93
 Chemical(s) Introduced into Chamber CO/CO₂
 Amount 7.5 liters CO; 3.0 liters CO₂; 1.0 liter air

Chamber Conditions before Introduction of Chemical
 Temperature 63.8°F
 Pressure 0.0 psig

Cell Conditions before Introduction of Chemical
 Temperature 55°F
 Pressure 608.2 mm Hg

Time of Day Chemicals Introduced into Chamber 10:20 am
 Time of Day Mixing Fan Turned on 10:21 am
 Sampling Information:

| | | | |
|-------------------|--------|-------------|----------|
| Sample No. | Blank | Time of Day | 9:45 am |
| Chamber T | 63.8°F | Chamber P | 0.0 psig |
| Flowrater Setting | N/A | Flowrater T | N/A |
| Sampling Time | N/A | | |

| | | | |
|-------------------|--------|-------------|----------|
| Sample No. | 1 | Time of Day | 10:26 am |
| Chamber T | 63.1°F | Chamber P | 0.8 psig |
| Flowrater Setting | N/A | Flowrater T | N/A |
| Sampling Time | N/A | | |

| | | | |
|-------------------|--------|-------------|----------|
| Sample No. | 2 | Time of Day | 10:32 am |
| Chamber T | 63.1°F | Chamber P | 0.6 psig |
| Flowrater Setting | N/A | Flowrater T | N/A |
| Sampling Time | N/A | | |

| | | | |
|-------------------|--------|-------------|----------|
| Sample No. | 3 | Time of Day | 10:37 am |
| Chamber T | 63.1°F | Chamber P | 0.6 psig |
| Flowrater Setting | N/A | Flowrater T | N/A |
| Sampling Time | N/A | | |

Notes: N/A = Not Available
 T = Temperature
 P = Pressure
 psig = pounds per square inch, gauge
 mm Hg = millimeters mercury

TABLE A-2. TEST OB DATA SHEET.

Date 4-27-93
 Chemical(s) Introduced into Chamber Aerozine-50
 Amount 4.0 cc (3.00 seconds, 0.0128-inch nozzle, $\Delta P = 31$ psig)

Chamber Conditions before Introduction of Chemical

Temperature 122.7°F
 Pressure 0.0 mm Hg

Cell Conditions before Introduction of Chemical

Temperature 69.2°F
 Pressure 612.9 mm Hg

Time of Day Chemicals Introduced into Chamber 3:08 pm and 15 seconds

Time of Day Mixing Fan Turned on 3:09 pm

Sampling Information:

| | | | |
|-------------------|--------------|-------------|----------|
| Sample No. | Blank | Time of Day | 3:01 pm |
| Chamber T | 122.7°F | Chamber P | 0.0 psig |
| Flowrater Setting | 6.0 | Flowrater T | 70.5°F |
| Sampling Time | 3:00 minutes | | |

| | | | |
|-------------------|--------------|-------------|----------|
| Sample No. | 1 | Time of Day | 3:22 pm |
| Chamber T | 115.8°F | Chamber P | 0.2 psig |
| Flowrater Setting | 6.0 | Flowrater T | 69.4°F |
| Sampling Time | 4:01 minutes | | |

| | | | |
|-------------------|--------------|-------------|----------|
| Sample No. | 2 | Time of Day | 3:45 pm |
| Chamber T | 109.5°F | Chamber P | 0.0 psig |
| Flowrater Setting | 6.0 | Flowrater T | 67.4°F |
| Sampling Time | 3:04 minutes | | |

| | | | |
|-------------------|--------------|-------------|----------|
| Sample No. | 3 | Time of Day | 5:15 pm |
| Chamber T | N/A | Chamber P | 0.0 psig |
| Flowrater Setting | 6.0 | Flowrater T | 66.0°F |
| Sampling Time | 3:07 minutes | | |

Notes: Sample 3 was taken after completion of primary test series. Mixing fan was off between samples 2 & 3, bubblers were not cooled nor previously cleaned.

N/A = Not Available
 mm Hg = millimeters mercury
 cc = cubic centimeters
 T = Temperature
 P = Pressure
 psig = pounds per square inch, gauge

TABLE A-3. TEST OC DATA SHEET.

Date 4-30-93
 Chemical(s) Introduced into Chamber N₂O₄
 Amount 5.5 cc

Chamber Conditions before Introduction of Chemical
 Temperature 115.4°F
 Pressure 0 psig

Cell Conditions before introduction of Chemical
 Temperature 64.4°F ambient
 Pressure 11.718 psia

Time of Day Chemicals Introduced into Chamber 10:28 am
 Time of Day Mixing Fan turned on 10:29 am
 Sampling Information:

| | | | |
|-------------------|---------|-------------|----------|
| Sample No. | 1 | Time of Day | 10:43 am |
| Chamber T | 109.7°F | Chamber P | 0 psig |
| Flowrater Setting | N/A | Flowrater T | N/A |
| Sampling Time | N/A | | |

| | | | |
|-------------------|---------|-------------|----------|
| Sample No. | 2 | Time of Day | 10:58 am |
| Chamber T | 106.2°F | Chamber P | 0 psig |
| Flowrater Setting | N/A | Flowrater T | N/A |
| Sampling Time | N/A | | |

Notes: N/A = Not Available
 cc = cubic centimeters
 psig = pounds per square inch, gauge
 psia = pounds per square inch, absolute
 T = Temperature
 P = Pressure

TABLE A-4. TEST 1 DATA SHEET.

Test No. 1 (Fuel-Rich)
Date: 4-23-93

N₂O₄ Supply Pressure 13 psig
A-50 Supply Pressure 39 psig
RP-1 Supply Pressure N/A
GOX Supply Pressure N/A

Chamber Conditions before Propellant Ignition
Temperature 73.7°F
Pressure 0 psig

Cell Conditions before Propellant Ignition
Temperature 69.6°F
Pressure 602.9 mm Hg

Time of Day that Ignition was Initiated 1:22 pm start sequence; 15 second delay to ignition after sequence start.

Solenoid "ON" Times N₂O₄ 2.0 seconds A-50 2.0 seconds
RP-1 N/A GOX N/A

Time of Day that Fan was Turned on 1:23 pm (after ignition)

| <u>Sample #</u> | <u>Type</u> | <u>Time of Day</u> | <u>Chamber</u> | | <u>Flowrater</u> | | |
|-----------------|-------------------------------|--------------------|----------------|---------------|------------------|--------------|-------------|
| | | | <u>Temp.</u> | <u>Press.</u> | <u>Setting</u> | <u>Temp.</u> | <u>Time</u> |
| Blank | Hoke Bottle | 12:31 pm | | 0.0 psig | N/A | N/A | N/A |
| Blank | N ₂ H ₄ | 12:31 pm | | 0.0 psig | 6.0 | 69.1°F | 3:00 min |
| Blank | Charcoal | 12:31 pm | | 0.0 psig | 6.0 | 68.9°F | 3:07 min |
| 1 | Hoke Bottle | 1:29 pm | | 0.0 psig | N/A | N/A | N/A |
| 1 | Charcoal | 1:32 pm | | 0.0 psig | 6.0 | 69.9°F | 3:13 min |
| 1 | N ₂ H ₄ | 1:36 pm | | 0.0 psig | 6.0 | 70.2°F | 3:01 min |
| 2 | Hoke Bottle | 1:54 pm | | 0.0 psig | N/A | N/A | N/A |
| 2 | Charcoal | 1:55 pm | | 0.0 psig | 6.0 | 71.1°F | 3:14 min |
| 2 | N ₂ H ₄ | 1:59 pm | | 0.0 psig | 6.0 | 71.0°F | 3:32 min |

Notes: N/A = Not Available
psig = pounds per square inch, gauge
mm Hg = millimeters mercury
min = minutes

TABLE A-5. TEST 2 DATA SHEET.

Test No. 2 (Oxidizer - Rich)
Date: 4-29-93

N₂O₄ Supply Pressure 116 psig
A-50 Supply Pressure 27 psig
RP-1 Supply Pressure N/A
GOX Supply Pressure N/A

Chamber Conditions before Propellant Ignition
Temperature 80.1°F
Pressure 0.1 psig

Cell Conditions before Propellant Ignition
Temperature on chart
Pressure 614.4 mm Hg

Time of Day that Ignition was Initiated 10:56 am and 0 seconds

Solenoid "ON" Times N₂O₄ 2 seconds A-50 2 seconds
RP-1 N/A GOX N/A

Time of Day that Fan was Turned on 10:56 am and 30 seconds; sequencer "ON"

| Sample # | Type | Time of Day | Chamber | | Flowrater | | Time |
|-----------------|----------|-------------|---------|----------|-----------|--------|----------|
| | | | Temp. | Press. | Setting | Temp. | |
| Chamber Gas | Blank | 10:35 am | | | | | |
| Charcoal | Blank | 10:41 am | | 0.0 psig | 6.0 | 65.9°F | |
| NO _x | Blank | 10:43 am | | | | | |
| NO _x | Sample 1 | 11:08 am | | 0.6 psig | | | |
| NO | Sample 1 | 11:14 am | | 0.2 psig | | | |
| Chamber Gas | Sample 1 | 11:16 am | | 0.0 psig | | | |
| Charcoal | Sample 1 | 11:17 am | | 0.0 psig | 6.0 | 68.6°F | 3:23 min |
| NO _x | Sample 2 | 11:26 am | | 0.0 psig | | | |
| NO | Sample 2 | 11:28 am | | 0.0 psig | | | |
| Chamber Gas | Sample 2 | 11:29 am | | 0.0 psig | | | |
| Charcoal | Sample 2 | 11:30 am | | 0.0 psig | 6.0 | 69.1°F | 3:05 min |

Notes: N/A = Not Available
Blanks in Table indicate unavailable data
psig = pounds per square inch, gauge
mm Hg = millimeters mercury
min = minutes

TABLE A-6. TEST 3 DATA SHEET.

Test No. 3 (Stoichiometric N₂O₄/A-50)

Date: 5-4-93

N₂O₄ Supply Pressure 52 psig

A-50 Supply Pressure 48 psig

RP-1 Supply Pressure

GOX Supply Pressure

Chamber Conditions before Propellant Ignition

Temperature 79.2°F

Pressure 0 psig

Cell Conditions before Propellant Ignition

Temperature 73.5°F

Pressure 604 mm Hg

Time of Day that Ignition was Initiated 11:35 am and 0 seconds

| | | | | |
|---------------------|-------------------------------|-----------|------|-----------|
| Solenoid "ON" Times | N ₂ O ₄ | 3 seconds | A-50 | 3 seconds |
| | RP-1 | | GOX | |

Time of Day that Fan was Turned on 11:35 am and 30 seconds

TABLE A-6. TEST 3 DATA SHEET (CONTINUED).

| <u>Sample #</u> | <u>Type</u> | <u>Time of Day</u> | <u>Chamber</u> | | <u>Flowrater</u> | | <u>Time</u> |
|-----------------|-------------------------------|--------------------|----------------|---------------|------------------|--------------|-------------|
| | | | <u>Temp.</u> | <u>Press.</u> | <u>Setting</u> | <u>Temp.</u> | |
| Blank | N ₂ H ₄ | 11:02 am | 79.2°F | 0.0 psig | 6.0 | 74.6°F | 3:04 min |
| Blank | Charcoal | 11:10 am | | 0.0 psig | 6.0 | 75.2°F | 3:00 min |
| Blank | Hoke Bottle | 11:14 am | | 0.0 psig | | | |
| Blank | NO _x | 11:20 am | | 0.0 psig | | | |
| Blank | NO | 11:20 am | | 0.0 psig | | | |
| Sample 1 | N ₂ H ₄ | 11:49 am | 83.6°F | 0.8 psig | 6.0 | 77.8°F | 3:04 min |
| Sample 1 | Charcoal | 11:53 am | 83.7°F | 0.7 psig | 6.0 | 78.0°F | 3:02 min |
| Sample 1 | Hoke Bottle | 11:57 am | 83.7°F | 0.7 psig | | | |
| Sample 1 | NO _x | 12:02 pm | 83.8°F | 0.6 psig | | | |
| Sample 1 | NO | 12:04 pm | | | | | |
| Sample 2 | N ₂ H ₄ | 12:08 pm | 83.9°F | 0.1 psig | 6.0 | 79.4°F | 3:12 min |
| Sample 2 | Charcoal | 12:11 pm | 83.9°F | 0.0 psig | 6.0 | 79.8°F | 3:12 min |
| Sample 2 | Hoke Bottle | 12:15 pm | 83.9°F | -0.1 psig | | | |
| Sample 2 | NO _x | 12:19 pm | | | | | |
| Sample 2 | NO | 12:21 pm | | | | | |

Notes: Blanks in Table indicate unavailable data
 psig = pounds per square inch, gauge
 mm Hg = millimeters mercury
 min = minutes

TABLE A-7. TEST 4 DATA SHEET.

Test No. 4 (Titan IV)
Date: 5/5/93

N₂O₄ Supply Pressure 52 psig
A-50 Supply Pressure 48 psig
RP-1 Supply Pressure
GOX Supply Pressure

Chamber Conditions before Propellant Ignition
Temperature 73.6°F
Pressure -0.1 psig

Cell Conditions before Propellant Ignition
Temperature 67.9°F
Pressure 605.0 mm Hg

Time of Day that Ignition was Initiated 1:31 pm and 0 seconds

Solenoid "ON" Times N₂O₄ 3 seconds: A-50 3 seconds
RP-1 GOX

Time of Day that Fan was Turned on 1:31 pm and 30 seconds

| Sample # | Type | Time of Day | Chamber | | Flowrater | | Time |
|----------|--------------------------------|-------------|---------|-----------|-----------|--------|----------|
| | | | Temp. | Press. | Setting | Temp. | |
| Blank | NO _x | 12:58 pm | 73.5°F | 0.0 psig | | | |
| Blank | N ₂ H ₄ | 1:00 pm | | -0.3 psig | 6.0 | 70.6°F | 3:03 min |
| Blank | Cl | 1:03 pm | 73.5°F | -0.5 psig | 6.0 | 71.3°F | 3:09 min |
| Blank | Charcoal | 1:07 pm | 73.5°F | -0.6 psig | 6.0 | 71.5°F | 3:02 min |
| Blank | Hoke Bottle | 1:13 pm | 73.5°F | | | | |
| Sample 1 | NO _x | 1:50 pm | 81.8°F | 0.1 psig | | | |
| Sample 1 | NO | 1:52 pm | 81.8°F | 0.1 psig | | | |
| Sample 1 | N ₂ H ₄ | 1:53 pm | 81.8°F | 0.1 psig | 6.0 | 70.9°F | 3:06 min |
| Sample 1 | Cl | 1:56 pm | 82.0°F | -0.1 psig | 6.0 | 70.8°F | 3:08 min |
| Sample 1 | Charcoal | 2:00 pm | 82.3°F | -0.2 psig | 6.0 | 70.3°F | 3:02 min |
| Sample 1 | Hoke Bottle | 2:05 pm | | | | | |
| Sample 2 | NO _x | 2:11 pm | 82.4°F | -0.5 psig | | | |
| Sample 2 | NO | 2:12 pm | 82.4°F | -0.5 psig | | | |
| Sample 2 | *N ₂ H ₄ | 2:14 pm | | | 6.0 | 69.7°F | 3:00 min |
| Sample 2 | **Cl | 2:17 pm | 82.5°F | -0.7 psig | 6.0 | 70.1°F | 3:02 min |
| Sample 2 | **Charcoal | 2:20 pm | | | 6.0 | 69.9°F | 3:00 min |
| Sample 2 | Hoke Bottle | 2:24 pm | 82.3°F | -0.9 psig | | | |

Notes: *hydrazine bubbler loose
**sampling time approximate
Blanks in Table indicate unavailable data
psig = pounds per square inch, gauge
mm Hg = millimeters mercury
min = minutes

TABLE A-8. TEST 5 DATA SHEET.

Test No. 5
Date: 5/7/93

N₂O₄ Supply Pressure N/A
A-50 Supply Pressure N/A
RP-1 Supply Pressure 80 psig
GOX Supply Pressure 182 psig

Chamber Conditions before Propellant Ignition
Temperature 70.3°F
Pressure 0 psig

Cell Conditions before Propellant Ignition
Temperature 69.6°F
Pressure 0 psig

Time of Day that Ignition was Initiated 1:40 p.m.

Solenoid "ON" Times N₂O₄ N/A A-50 N/A
RP-1 2 seconds GOX 2 seconds

Time of Day that Fan was Turned on 1:40 p.m.

| <u>Sample #</u> | <u>Type</u> | <u>Time of Day</u> | <u>Chamber</u> | | <u>Flowrater</u> | | <u>Time</u> |
|-----------------|-------------|--------------------|----------------|---------------|------------------|--------------|-------------|
| | | | <u>Temp.</u> | <u>Press.</u> | <u>Setting</u> | <u>Temp.</u> | |
| Blank | Charcoal | 1:35 pm | 70.5°F | 0.0 psig | 6 | 70°F | 3 min |
| Blank | Hoke Bottle | 1:35 pm | 70.5°F | 0.0 psig | N/A | N/A | N/A |
| Sample 1 | Charcoal | 1:55 pm | 79.5°F | 0.5 psig | 6 | 69.4°F | 3 min |
| Sample 1 | Hoke Bottle | 1:55 pm | 79.5°F | 0.5 psig | N/A | N/A | N/A |
| Sample 2 | Charcoal | 2:10 pm | 74.9°F | 0.2 psig | 6 | N/A | 3 min |
| Sample 2 | Hoke Bottle | 2:10 pm | 74.9°F | 0.2 psig | N/A | N/A | N/A |

Notes: N/A = Not Available
psig = pounds per square inch, gauge
min = minutes

TABLE A-9. TEST 6 DATA SHEET.

Test No. 6
Date: 5-11-93

N₂O₄ Supply Pressure N/A
A-50 Supply Pressure N/A
RP-1 Supply Pressure 19 psig
GOX Supply Pressure 520 psig

Chamber Conditions before Propellant Ignition

Temperature 75.1°F
Pressure 0 psig

Cell Conditions before Propellant Ignition

Temperature 67.2°F
Pressure 616.1 mm Hg

Time of Day that Ignition was Initiated 1:55 pm and 0 seconds

| | | | | |
|---------------------|-------------------------------|-----------|------|-----------|
| Solenoid "ON" Times | N ₂ O ₄ | N/A | A-50 | N/A |
| | RP-1 | 2 seconds | GOX | 2 seconds |

Time of Day that Fan was Turned on 1:55 pm and 30 seconds

| <u>Sample #</u> | <u>Type</u> | <u>Time of Day</u> | <u>Chamber</u> | | <u>Flowrater</u> | | <u>Time</u> |
|-----------------|-------------|--------------------|----------------|---------------|------------------|--------------|------------------------|
| | | | <u>Temp.</u> | <u>Press.</u> | <u>Setting</u> | <u>Temp.</u> | |
| Blank | Hoke Bottle | 1:30 pm | 75.1°F | -0.1 psig | | | |
| Blank | Charcoal | 1:37 pm | 75.0°F | -0.1 psig | | | 1.0 liter thru syringe |
| Sample 1 | Hoke Bottle | 2:11 pm | 75.4°F | 0.9 psig | | | |
| Sample 1 | Charcoal | 2:14 pm | 75.4°F | 0.9 psig | | | 1.0 liter thru syringe |
| Sample 2 | Hoke Bottle | 2:26 pm | 75.5°F | 0.7 psig | | | |
| Sample 2 | Charcoal | 2:30 pm | 75.5°F | 0.7 psig | | | 1.0 liter thru syringe |

Notes: N/A = Not Available
Blanks in Table indicate unavailable data
psig = pounds per square inch, gauge
mm Hg = millimeters mercury
min = minutes

TABLE A-10. TEST 7 DATA SHEET.

Test No. 7
Date: 5/13/93

N₂O₄ Supply Pressure N/A
A-50 Supply Pressure N/A
RP-1 Supply Pressure 20 psig
GOX Supply Pressure 436 psig

Chamber Conditions before Propellant Ignition
Temperature 79.3°F
Pressure 0 psig

Cell Conditions before Propellant Ignition
Temperature 67.7°F
Pressure 615.3 mm Hg

Time of Day that Ignition was Initiated 9:00 am and 0 seconds

Solenoid "ON" Times N₂O₄ N/A A-50 N/A
RP-1 4 seconds GOX 4 seconds

Time of Day that Fan was Turned on 9:00 am and 30 seconds

| <u>Sample #</u> | <u>Type</u> | <u>Time of Day</u> | <u>Chamber</u> | | <u>Flowrater</u> | | <u>Time</u> |
|-----------------|-------------|--------------------|----------------|---------------|------------------|--------------|-------------|
| | | | <u>Temp.</u> | <u>Press.</u> | <u>Setting</u> | <u>Temp.</u> | |
| Blank | Hoke Bottle | 8:35 am | 79.8°F | 0.0 psig | N/A | N/A | N/A |
| Blank | Charcoal | 8:39 am | 79.8°F | 0.0 psig | 6.0 | 68.7°F | 3:07 min |
| Sample 1 | Hoke Bottle | 9:15 am | 82.5°F | 1.2 psig | N/A | N/A | N/A |
| Sample 1 | Charcoal | 9:16 am | 82.5°F | 1.1 psig | 6.0 | 68.3°F | 3:17 min |
| Sample 2 | Hoke Bottle | 9:30 am | 82.3°F | 1.0 psig | N/A | N/A | N/A |
| Sample 2 | Charcoal | 9:31 am | 82.3°F | 1.0 psig | 6.0 | 68.5°F | 3:01 min |

Notes: N/A = Not Available
psig = pounds per square inch, gauge
mm Hg = millimeters mercury
min = minutes

TABLE A-11. TEST 8 DATA SHEET.

Test No. 8
Date: 5/20/93

N₂O₄ Supply Pressure 52 psig
A-50 Supply Pressure 48 psig
RP-1 Supply Pressure 35 psig
GOX Supply Pressure 578 psig

Chamber Conditions before Propellant Ignition

Temperature 58.9°F
Pressure 0 psig

Cell Conditions before Propellant Ignition

Temperature 60.1°F
Pressure 610.9 mm Hg

Time of Day that Ignition was Initiated 10:44 am and 0 seconds

| | | | | |
|---------------------|-------------------------------|-----------|------|-----------|
| Solenoid "ON" Times | N ₂ O ₄ | 3 seconds | A-50 | 3 seconds |
| | RP-1 | 3 seconds | GOX | 3 seconds |

Time of Day that Fan was Turned on 10:45 am and 0 seconds

TABLE A-11. TEST 8 DATA SHEET (CONTINUED).

| <u>Sample #</u> | <u>Type</u> | <u>Time of Day</u> | <u>Chamber</u> | | <u>Flowrater</u> | | <u>Time</u> |
|-----------------|-------------------------------|--------------------|----------------|---------------|------------------|--------------|-------------|
| | | | <u>Temp.</u> | <u>Press.</u> | <u>Setting</u> | <u>Temp.</u> | |
| Blank | Hoke Bottle | 10:11 am | 58.9°F | 0.0 psig | | | |
| Blank | NO _x | 10:17 am | 59.0°F | -0.2 psig | | | |
| Blank | Charcoal | 10:19 am | 59.1°F | -0.2 psig | 6.0 | 60.6°F | 3:00 min |
| Blank | N ₂ H ₄ | 10:23 am | 59.2°F | -0.3 psig | 6.0 | 60.8°F | 3:04 min |
| Blank | Cl | 10:26 am | 59.2°F | -0.4 psig | 6.0 | 61.3°F | 3:03 min |
| Sample 1 | Hoke Bottle | 11:00 am | 68.8°F | 1.3 psig | | | |
| Sample 1 | NO _x | 11:05 am | 69.1°F | 1.0 psig | | | |
| Sample 1 | Charcoal | 11:07 am | 69.3°F | 0.9 psig | 6.0 | 64.0°F | 3:29 min |
| Sample 1 | N ₂ H ₄ | 11:11 am | 69.5°F | 0.7 psig | 6.0 | 64.0°F | 3:01 min |
| Sample 1 | Cl | 11:15 am | 69.7°F | 0.6 psig | 6.0 | 64.0°F | 3:01 min |
| Sample 2 | Hoke Bottle | 11:18 am | | | | | |
| Sample 2 | NO _x | 11:24 am | 70.1°F | 0.3 psig | | | |
| Sample 2 | Charcoal | 11:27 am | 70.1°F | 0.2 psig | 6.0 | 65.0°F | 3:01 min |
| Sample 2 | N ₂ H ₄ | 11:30 am | 70.2°F | 0.1 psig | 6.0 | 66.0°F | 3:03 min |
| Sample 2 | Cl | 11:34 am | 70.3°F | 0 psig | 6.0 | 66.0°F | 3:02 min |

Notes: Blanks in Table indicate unavailable data
 psig = pounds per square inch, gauge
 mm Hg = millimeters mercury
 min = minutes

TABLE A-12. TEST 9 DATA SHEET.

Test No. 9
Date: 5/14/93

N₂O₄ Supply Pressure N/A
A-50 Supply Pressure N/A
RP-1 Supply Pressure N/A
GOX Supply Pressure N/A

Chamber Conditions before Propellant Ignition

Temperature N/A
Pressure 0 psig

Cell Conditions before Propellant Ignition

Temperature 78.0°F
Pressure 602 mm Hg

Time of Day that Ignition was Initiated 9:15 am and 0 seconds

| | | | | |
|---------------------|-------------------------------|-----|------|-----|
| Solenoid "ON" Times | N ₂ O ₄ | N/A | A-50 | N/A |
| | RP-1 | N/A | GOX | N/A |

Time of Day that Fan was Turned on 9:15 am and 30 seconds

| <u>Sample #</u> | <u>Type</u> | <u>Time of Day</u> | <u>Chamber</u> | | <u>Flowrater</u> | | <u>Time</u> |
|-----------------|-------------|--------------------|----------------|---------------|------------------|--------------|-------------|
| | | | <u>Temp.</u> | <u>Press.</u> | <u>Setting</u> | <u>Temp.</u> | |
| Blank | Hoke Bottle | 8:50 am | 78.0°F | 0.0 psig | | | |
| Blank | Cl | 8:55 am | | | 6.0 | 74.1°F | 3:15 min |
| Blank | Charcoal | 8:57 am | | | 6.0 | 74.8°F | 3:12 min |
| Sample 1 | Hoke Bottle | 9:30 am | 82.4°F | 0.4 psig | | | |
| Sample 1 | Cl | 9:32 am | 82.7°F | 0.3 psig | 6.0 | 76.6°F | 3:08 min |
| Sample 1 | Charcoal | 9:35 am | 82.9°F | 0.2 psig | 6.0 | 76.8°F | 3:34 min |
| Sample 2 | Hoke Bottle | 9:45 am | 83.3°F | 0.1 psig | | | |
| Sample 2 | Cl | 9:46 am | 83.4°F | 0.0 psig | 6.0 | 77.2°F | 3:04 min |
| Sample 2 | Charcoal | 9:50 am | 83.6°F | -0.1 psig | 6.0 | 77.5°F | 3:02 min |

Notes: N/A = Not Available
Blanks in Table indicate unavailable data
psig = pounds per square inch, gauge
mm Hg = millimeters mercury
min = minutes

TABLE A-13. TEST 10 DATA SHEET.

Test No. 10
Date: 5/17/93

N₂O₄ Supply Pressure N/A
A-50 Supply Pressure N/A
RP-1 Supply Pressure N/A
GOX Supply Pressure N/A

Chamber Conditions before Propellant Ignition
Temperature 63.2°F
Pressure 0 psig

Cell Conditions before Propellant Ignition
Temperature 62.3°F
Pressure 611.1 mm Hg

Time of Day that Ignition was Initiated 10:10 am and 0 seconds

Solenoid "ON" Times N₂O₄ N/A A-50 N/A
RP-1 N/A GOX N/A

Time of Day that Fan was Turned on 10:10 am and 30 seconds

| <u>Sample #</u> | <u>Type</u> | <u>Time of Day</u> | <u>Chamber</u> | | <u>Flowrater</u> | | <u>Time</u> |
|-----------------|-------------|--------------------|----------------|---------------|------------------|--------------|-------------|
| | | | <u>Temp.</u> | <u>Press.</u> | <u>Setting</u> | <u>Temp.</u> | |
| Blank | Hoke Bottle | 9:50 am | 63.2°F | 0 psig | N/A | N/A | N/A |
| Blank | Charcoal | 9:52 am | 63.2°F | 0 psig | 6.0 | 60.6°F | 3:05 min |
| Blank | Cl | 9:56 am | 63.2°F | -0.2 psig | 6.0 | 61.2°F | 3:16 min |
| Sample 1 | Hoke Bottle | 10:26 am | 66.7°F | 0.4 psig | N/A | N/A | N/A |
| Sample 1 | Charcoal | 10:28 am | 66.9°F | 0.3 psig | 6.0 | 63.0°F | 3 min** |
| Sample 1 | Cl | N/A | 67.1°F | 0.2 psig | 6.0 | 63.2°F | 3:16 min |
| Sample 2 | Hoke Bottle | 10:40 am | 67.4°F | 0 psig | N/A | N/A | N/A |
| Sample 2 | Charcoal | 10:42 am | 67.4°F | 0 psig | 6.0 | 63.3°F | 3:12 min |
| Sample 2 | Cl | 10:45 am | 67.5°F | -0.1 psig | 6.0 | 63.4°F | 3:04 min |

Notes: **Approximate time
N/A = Not Available
psig = pounds per square inch, gauge
mm Hg = millimeters mercury
min = minutes

TABLE A-14. TEST 11 DATA SHEET.

Test No. 11
Date: 5/18/93

N₂O₄ Supply Pressure N/A
A-50 Supply Pressure N/A
RP-1 Supply Pressure 19 psig
GOX Supply Pressure 520 psig

Chamber Conditions before Propellant Ignition
Temperature 59.4°F
Pressure 0 psig

Cell Conditions before Propellant Ignition
Temperature 63.2°F
Pressure 612.7 mm Hg

Time of Day that Ignition was Initiated 10:16 am and 0 seconds

Solenoid "ON" Times N₂O₄ N/A A-50 N/A
RP-1 2 seconds GOX 2 seconds

Time of Day that Fan was Turned on 10:16 am and 30 seconds

| <u>Sample #</u> | <u>Type</u> | <u>Time of Day</u> | <u>Chamber</u> | | <u>Flowrater</u> | | <u>Time</u> |
|-----------------|-------------|--------------------|----------------|---------------|------------------|--------------|-------------|
| | | | <u>Temp.</u> | <u>Press.</u> | <u>Setting</u> | <u>Temp.</u> | |
| Blank | Hoke Bottle | 9:50 am | 59.4°F | 0.0 psig | N/A | N/A | N/A |
| Blank | Charcoal | 9:57 am | 59.3°F | 0.0 psig | 6.0 | 61.6°F | 3:06 min |
| Sample 1 | Hoke Bottle | 10:31 am | 60.9°F | 0.9 psig | N/A | N/A | N/A |
| Sample 1 | Charcoal | 10:33 am | 61.0°F | 0.9 psig | 6.0 | 64.0°F | 3:17 min |
| Sample 2 | Hoke Bottle | 10:45 am | 61.5°F | 0.8 psig | N/A | N/A | N/A |
| Sample 2 | Charcoal | 10:47 am | 61.6°F | 0.7 psig | 6.0 | 64.6°F | 3:09 min* |

Notes: *Teflon tube fitting loose
N/A = Not Available
psig = pounds per square inch, gauge
mm Hg = millimeters mercury
min = minutes

APPENDIX B
CHAMBER SCHEMATIC

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TEST CHAMBER SCHEMATIC

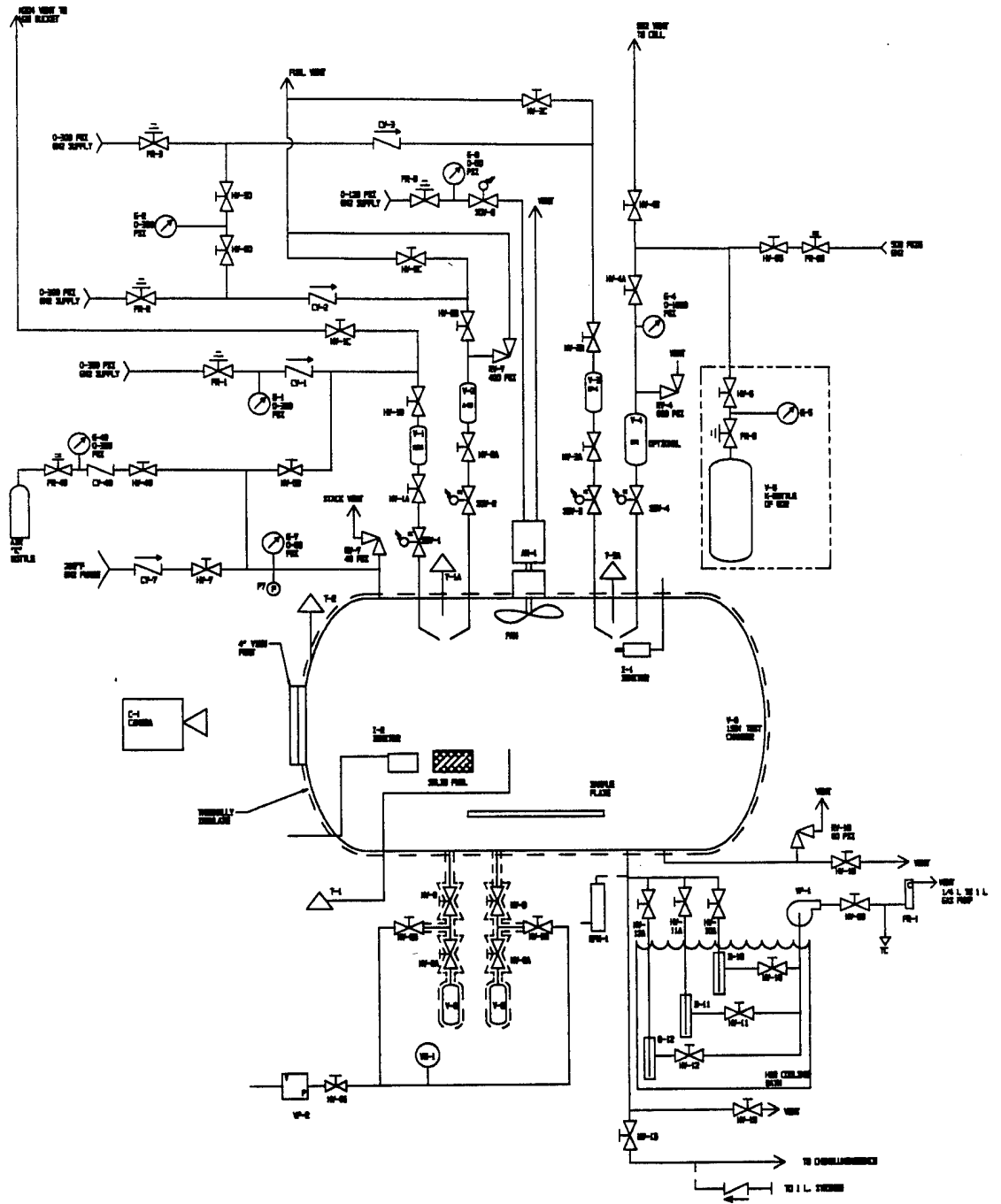


Figure B-1. Test Chamber Schematic.

APPENDIX C
DETERMINATION OF NOZZLE DELIVERY PRESSURES

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TABLE C-1. CALCULATION OF NOZZLE DELIVERY PRESSURE, AEROZINE-50.

$$\Delta P = \left(\frac{\dot{m}}{CA} \right)^2 \left(\frac{144 \text{ in}^2/\text{ft}^2}{2\rho g_c} \right)$$

\dot{m} = desired flow rate, lb/s

C = nozzle discharge coefficient, unitless

A = nozzle area, in²

ρ = density of A-50 = 0.8987 g/cm³ = 56.11 lb/ft³

g_c = 32.2 ft/s²

ΔP = delivery pressure, psig

| Test | A-50 vol, cm ³ | Time, s | \dot{m} , lb/s | Orifice Size, in. | C | A, in ² | ΔP , psig |
|------|------------------------------|------------|---------------------|----------------------|--------|--------------------------|----------------------|
| 0 | 4.0 | 3 | 0.00264 | 0.0128 | 0.7456 | 1.287 x 10 ⁻⁴ | 30.2 |
| 1 | 5.0 | 2 | 0.00495 | 0.018 | 0.631 | 2.545 x 10 ⁻⁴ | 37.9 |
| 2 | 2.5 | 2 | 0.0025 | 0.0128 | 0.7456 | 1.287 x 10 ⁻⁴ | 27.0 |
| 3 | 5.0 | 3 | 0.0033 | 0.0128 | 0.7456 | 1.287 x 10 ⁻⁴ | 47.2 |
| 4 | 5.0 | 3 | 0.0033 | 0.128 | 0.7456 | 1.287 x 10 ⁻⁴ | 47.2 |
| 8 | 5.0 | 3 | 0.0033 | 0.128 | 0.7456 | 1.287 x 10 ⁻⁴ | 47.2 |

TABLE C-2. CALCULATION OF NOZZLE DELIVERY PRESSURE, N₂O₄.

$$\Delta P = \left(\frac{\dot{m}}{CA} \right)^2 \left(\frac{144 \text{ in}^2/\text{ft}^2}{2g_c} \right)$$

\dot{m} = desired flow rate, lb/s

C = nozzle discharge coefficient, unitless

A = nozzle area, in²

ρ = density of N₂O₄ = 1.433 g/cm³ = 89.46 lb/ft³

g_c = 32.2 ft/s²

ΔP = delivery pressure, psig

| Test | N ₂ O ₄ vol, cm ³ | Time, s | \dot{m} lb/s | Orifice Size, in. | C | A, in ² | ΔP , psig |
|------|---|------------|-------------------|----------------------|--------|--------------------------|----------------------|
| 0 | 5.5 | 3 | 0.00579 | 0.018 | 0.631 | 2.545 x 10 ⁻⁴ | 32.5 |
| 1 | 1.4 | 2 | 0.00221 | 0.0128 | 0.7414 | 1.287 x 10 ⁻⁴ | 13.4 |
| 2 | 6.9 | 2 | 0.0109 | 0.018 | 0.631 | 2.545 x 10 ⁻⁴ | 115.1 |
| 3 | 6.9 | 3 | 0.00727 | 0.018 | 0.631 | 2.545 x 10 ⁻⁴ | 51.2 |
| 4 | 6.9 | 3 | 0.00727 | 0.018 | 0.631 | 2.545 x 10 ⁻⁴ | 51.2 |
| 8 | 6.9 | 3 | 0.00727 | 0.018 | 0.631 | 2.545 x 10 ⁻⁴ | 51.2 |

TABLE C-3. CALCULATION OF NOZZLE DELIVERY PRESSURE, RP-1.

$$\Delta P = \left(\frac{\dot{m}}{CA} \right)^2 \left(\frac{144 \text{ in}^2/\text{ft}^2}{2\rho g_c} \right)$$

- \dot{m} = desired flow rate, lb/s
- C = nozzle discharge coefficient, unitless
- A = nozzle area, in²
- ρ = density of RP-1 = 46.64 lb/ft³
- g_c = 32.2 ft/s²
- ΔP = delivery pressure, psig

| Test | RP-1 vol, cm ³ | Time, s | \dot{m} , lb/s | Orifice Size, in. | C | A, in ² | ΔP , psig |
|------|------------------------------|------------|---------------------|----------------------|--------|---------------------------|----------------------|
| 5 | 5.1 | 2 | 0.00420 | 0.0135 | 0.7212 | 1.4314 x 10 ⁻⁴ | 79.3 |
| 6 | 2.5 | 2 | 0.00206 | 0.0135 | 0.7212 | 1.4314 x 10 ⁻⁴ | 19.1 |
| 7 | 5.1 | 4 | 0.00210 | 0.0135 | 0.7212 | 1.4314 x 10 ⁻⁴ | 19.8 |
| 8 | 5.1 | 3 | 0.00280 | 0.0135 | 0.7212 | 1.4314 x 10 ⁻⁴ | 35.3 |

TABLE C-4. CALCULATION OF NOZZLE DELIVERY PRESSURE, GOX.

$$W_{\max} = CA_2 P_1 \sqrt{g_c k \left(\frac{M}{RT_1} \right) \left(\frac{2}{k+1} \right)^{\frac{k+1}{k-1}}} = CA_2 P_1 (0.02434)$$

$$R = 1545 \frac{\text{ft}\cdot\text{lb}}{\text{R}\cdot\text{lb mole}}$$

C = discharge coefficient = 0.6294 (0.0312-in. diameter nozzle), unitless

A₂ = Orifice Area = πD²/4

P₁ = Upstream Pressure, psig

g_c = 32.2 lb_mft/lbf s²

k = 1.401

M = 32 lb/lb-mole

T = 68°F + 460 = 528°R

| Test | O ₂ vol, scc | Time, s | \dot{m} lb/s | Orifice Size, in. | A ₂ , in ² | P ₁ , psig | ΔP, psig |
|------|-------------------------|---------|----------------|-------------------|----------------------------------|-----------------------|----------|
| 5 | 1263 | 2 | 0.00199 | 0.0312 | 7.645 x 10 ⁻⁴ | 169.8 | 182 |
| 6 | 6317 | 2 | 0.00995 | 0.040 | 1.257 x 10 ⁻³ | 516.7 | 528 |
| 7 | 6317 | 4 | 0.00497 | 0.0312 | 7.645 x 10 ⁻⁴ | 424.7 | 436 |
| 8 | 6317 | 3 | 0.00663 | 0.0312 | 7.645 x 10 ⁻⁴ | 566.2 | 578 |

APPENDIX D
SAMPLE ANALYSIS PLAN

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SAMPLE ANALYSIS PLAN

This document outlines sampling requirements and the specific approach for analysis of samples taken during the Launch Vehicle Abort propellant interaction tests.

A. DATA QUALITY

Data quality information was provided which was used to quantitate the precision and accuracy of selected analytical methodologies employed for this project. This information is defined below and was designed to meet the data quality objectives of this project.

| <u>Quality Controls</u> | <u>Assessment</u> |
|-------------------------------|----------------------|
| Method Blank | Method Contamination |
| Control Standard Recovery | Method Accuracy |
| Replicate Standard Deviation* | Method Precision |

* - whenever applicable

Based on historical information from routine hydrazine analyses at the Chemical Technology Laboratory (CTL), the results for hydrazine data are estimated to have the following uncertainty:

| | |
|-----------|------------------------------------|
| Hydrazine | $\pm 13\%$ at 99% Confidence Limit |
| | $\pm 11\%$ at 95% Confidence Limit |
| UDMH | $\pm 20\%$ at 99% Confidence Limit |
| | $\pm 19\%$ at 95% Confidence Limit |

It is expected that these limits also applied to samples analyzed for this project.

There is no CTL data quality history available for the other analytes which were quantified for this project.

B. SAMPLE REQUIREMENTS

Samples from this test involved the use of several types of sampling devices. The following describes the sampling requirements and handling of samples received at CTL.

1. Hoke Bottles

Samples are to be delivered to CTL as soon as possible after the sampling operation is complete. Bottles and sampling syringes to be used are to be placed in an oven set at nominal 50°C immediately upon receipt at CTL. Allow samples to equilibrate for 30 minutes before analysis. Analyses for CH₄, NH₃, and H₂ must occur as soon as possible after the 30 minute equilibration time. The time at which actual analyses for these three species occurs will be recorded. Bottles and sampling syringes are to be maintained at 50°C until analysis is complete.

Analytes: CO, CO₂, CH₄
H₂, O₂
NH₃

2. 2 Percent HCl Bubbler

Bubbler must be maintained in an ice bath or packed in ice during sampling to maximize efficiency. Upon receipt at CTL, ensure that inlet and outlet of bubbler are capped. Check pH of solution with an indicator strip. If pH is not <2 , add a few drops of concentrated HCl and re-check pH.

Note: If initial pH was not <2 , analysis results for hydrazine and UDMH may not be representative of actual concentrations in the test chamber. This bubbler containing 20 milliliters of 2 percent HCl will have a collection capacity of approximately 6.2 standard liters of 1.3 volume percent A-50 vapor.

Secure bubbler in an upright position in a refrigerator at $4 \pm 2^{\circ}\text{C}$ until analysis.

Analytes: Hydrazine and UDMH

3. 0.2N NaOH Bubbler

Bubbler must be maintained in an ice bath or packed in ice during sampling to maximize efficiency. Upon receipt at CTL, ensure that inlet and outlet of bubbler are capped. Check pH of solution with an indicator strip. If pH is not >9 , add a few drops of 30 percent NaOH solution and re-check pH. Secure bubbler in an upright position in a refrigerator at $4 \pm 2^{\circ}\text{C}$ until analysis.

Note: If initial pH was not >9 , analysis result for HCl may not be representative of actual concentration in the test chamber. This bubbler containing 20 milliliters of 0.2N NaOH will have a collection capacity of approximately 6.4 standard liters of 1 volume percent HCl vapor.

Analyte: HCl as chloride

4. Charcoal Tube

The tube must be maintained in an ice bath or packed in ice during sampling. Upon receipt at CTL, ensure that both ends of the tube are securely capped. Place tube in a refrigerator at $4 \pm 2^{\circ}\text{C}$ until analysis.

Analytes: Gas phase organics

5. Witness Plate 1 (Organics)

Witness plate will be covered immediately on removal from test chamber and transported to CTL as soon as possible. Upon receipt at CTL, remove the protective cover from the plate. Carefully rinse the entire inner surface of the plate with approximately 90 milliliters of methylene chloride solvent and collect the rinse. Transfer the rinse to a 100 milliliter volumetric flask and fill to the mark with clean methylene chloride. Invert flask to mix.

Analytes: Condensed organics (GC/MS and FTIR)

6. Witness Plate 2 (Inorganics)

Witness plate will be covered immediately on removal from test chamber and transported to CTL as soon as possible. Upon receipt at CTL, remove the protective cover from the plate. Carefully rinse the entire inner surface of the plate with approximately 90 milliliters of deionized and distilled water (DIDW) or reagent water and collect the rinse. Transfer the rinse to a 100 milliliter volumetric flask and fill to the mark with DIDW or reagent water. Invert flask to mix. Divide this sample into separate aliquots for specific analyses as follows:

N_2H_4 /UDMH - Transfer 20.0 milliliters of the rinse solution to a clean bottle and add 0.2 milliliters of concentrated HCl. Mix gently. Verify that pH is <2 and add additional concentrated HCl dropwise if necessary. Store in a refrigerator at $4 \pm 2^\circ C$ until analysis.

HCl- Transfer 20.0 milliliters of the solution to a clean Nalgene® bottle. Add a few drops of 30% NaOH solution until a pH of >9 is achieved. Store in a refrigerator at $4 \pm 2^\circ C$ until analysis.

pH/ HNO_3 - Save the remaining 60.0 milliliters of solution for these measurements. Store in a refrigerator at $4 \pm 2^\circ C$ until analysis.

C. ANALYSIS APPROACH

1. Carbon Monoxide, Carbon Dioxide, and Methane

| | | |
|--------------------|---|--|
| Analyte(s) | : | CO, CO ₂ , CH ₄ |
| Collection Device | : | Hoke Bottle |
| Analytical Method | : | GC/Methanizer/FID (Quantitative) |
| Sampling Technique | : | Gas-Tight Syringe |
| Standard(s) | : | #1 - 0.1% each CO, CO ₂ , CH ₄ in N ₂ #2 - 0.3% each CO, CO ₂ , CH ₄ in N ₂ #3 - 1.0% each CO, CO ₂ , CH ₄ in N ₂ #4 - 3.0% each CO, CO ₂ , CH ₄ in N ₂ #5 - 5.0% each CO, CO ₂ , CH ₄ in N ₂ |

Note: In these analyses, the concentration of standards which are prepared in the lab are nominal starting levels. Additional standards may be prepared as required.

Comments: After initial calibration and analysis of a blank (pure nitrogen), each sample will be analyzed three times. Each reported result will be the average of the three analyses. After the final sample analysis, the mid-range standard will be re-analyzed as a control standard. No more than nine analyses (three samples) will be performed between calibration and the control standard. If more than nine sample analyses are to be performed, an additional control standard will be run after each ninth analysis or less. Results will be reported as volume (molar) percent.

2. Hydrogen and Oxygen

| | | |
|--------------------|---|--|
| Analyte(s) | : | H ₂ , O ₂ |
| Collection Device | : | Hoke Bottle |
| Analytical Method | : | GC/TCD (Quantitative) |
| Sampling Technique | : | Gas-Tight Syringe |
| Standard(s) | : | #1 - 0.25% H ₂ in N ₂ #2 - 1.0% H ₂ in N ₂ #3 - 4.0% H ₂ in N ₂ #4 - 15% O ₂ in N ₂ #5 - 20% O ₂ in N ₂ #6 - 25% O ₂ in N ₂ |

Note: Due to safety concerns, standards containing percent levels of both oxygen and hydrogen are not available.

Comments: Sample analysis will consist of calibration, blank (pure nitrogen), sample analysis, and analysis of control standards which will consist of the two mid-range calibration standards.

3. AMMONIA

| | | |
|--------------------|---|---|
| Analyte(s) | : | NH ₃ |
| Collection Device | : | Hoke Bottle |
| Analytical Method | : | GC/TCD (Quantitative) |
| Sampling Technique | : | Gas-Tight Syringe |
| Standard(s) | : | #1 - 0.1% NH ₃ in N ₂ #2 - 0.5% NH ₃ in N ₂ #3 - 1.0% NH ₃ in N ₂ |

Comments: Sample analysis will consist of calibration, blank (pure nitrogen), sample analysis, and analysis of a control standard which will be the mid-range calibration standard.

4. HYDRAZINE and UDMH

| | | |
|-------------------|---|---|
| Analyte(s) | : | Hydrazine, UDMH (gas-phase and condensed- phase) |
| Collection Device | : | Bubbler containing 20.0 milliliters dilute acid solution for gas phase and witness plate 2 for condensed phase |
| Analytical Method | : | Furaldehyde GC/TSD (Quantitative) Standard CTL Method for trace analysis of hydrazine and UDMH |
| Standard(s) | : | #1 - 16 ug/L UDMH, 20 ug/L hydrazine #2 - 32 ug/L UDMH, 40 ug/L hydrazine #3 - 80 ug/L UDMH, 100 ug/L hydrazine |

Comments: Sample solutions will be diluted as necessary to place them in the range of the calibration curve. Sample analysis will consist of calibration, a blank, a laboratory control standard, triplicate sample analyses (from same dilutions), and a final check standard. Each reported sample result will be the average of the three analyses. Results for the gas phase sample will be converted to volume units (parts per million or percent) in the original gas sample based on volume of gas sample taken. Results for the witness plate rinse sample will be converted to total ug/unit area of witness plate.

5. HYDROGEN CHLORIDE

Analyte(s) : Hydrogen Chloride (gas-phase & condensed-phase)
Collection Device : Bubbler containing 20.0 milliliters dilute NaOH solution for gas-phase HCl and witness plate 2 for condensed-phase
Analytical Method : As Cl by silver nitrate titration (quantitative)
Standard(s) : Sodium Chloride primary standard used to prepare standards containing 1, 3, and 5 milligrams Cl⁻

Comments: It is suggested that at least 1 liter of gas-phase sample be collected for this analysis. Analysis of the condensed phase HCl will be done using an aliquot of the rinse water from the aqueous rinsed witness plate. Analysis procedure will consist of calibration, blank, sample analysis, and analysis of a control standard which will be the mid-range standard.

6. ACIDITY

Analyte(s) : pH
Collection Device : Witness plate 2
Analytical Method : pH meter
Standard(s) : Commercial standardized buffer solutions
#1 - pH 2.00 buffer
#2 - pH 4.00 buffer
#3 - pH 5.00 buffer
#4 - pH 7.00 buffer
#5 - pH 9.00 buffer
#6 - pH 10.00 buffer

Comments: An initial pH of the witness plate rinse solution will be determined using a pH test strip. The pH meter will then be calibrated using two standard buffer solutions which bracket the pH result obtained with the test strip. The pH of the rinse solution will then be determined using the pH meter. Finally, the pH meter will be checked using the buffer solution which is closest to the rinse solution.

7. GAS PHASE ORGANICS

Analyte(s) : Gas Phase Organics
Collection Device : Activated charcoal tube
Analytical Method : GC/MS (Qualitative)
Standard(s) : None

Comments: This procedure will involve methylene chloride or carbon disulfide extraction of the activated charcoal from the tube. The extract will then be analyzed by GC/MS in order to look for unforeseen organic compounds. Nitrosodimethylamine (NDMA) will also be qualitatively determined by this method. The extract of an unused charcoal tube will be analyzed as a blank. The presence of RP-1 in the sample may be a significant interferent in this analysis.

8. CONDENSED ORGANICS

Analyte(s) : Condensed Organics
Collection Device : Witness plate 1
Analytical Method : GC/MS and FTIR (Qualitative)
Standard(s) : None

Comments: This analysis will be performed on the methylene chloride rinse from the organics witness plate. The rinse will be analyzed for unforeseen organics by GC/MS and FTIR. NDMA will also be qualitatively determined from GC/MS data. An aliquot of the pre-test rinse solution will be analyzed as a blank. The presence of RP-1 in the sample may be a significant interferent in this analysis.

9. NITRIC ACID

Analyte(s) : Nitric Acid (HNO₃)
Collection Device : Witness plate 2
Analytical Method : As nitrate by spectrophotometry (Quantitative)
Standard(s) : Potassium Nitrate primary standard

Comments: This analysis will be performed at the Environmental Operations Laboratory (EOL) using a Technicon® Traacs 800 Spectrophotometer and standard EOL methodology. The test sample will be an aliquot of the water rinse of witness plate 2.

10. NITRIC OXIDE AND NITROGEN DIOXIDE

Analyte(s) : NO, NO₂
Collection Device : None (On-line analysis)
Analytical Method : Chemiluminescence
Standard(s) : 500 ppm NO in nitrogen

Comments: This analysis is done at the test site using a NO, NO_x chemiluminescent analyzer directly interfaced to the chamber. Readings will be taken when the chamber has cooled to 50°C. The sampling interface line will be maintained at 50°C or higher. Calibration will be performed prior to sampling using the 500 parts per million NO standard with the instrument set in the NO mode. After calibration, verification of NO₂ analysis will be performed using a 2500 parts per million NO₂ control standard with the instrument set in the NO_x mode. If the measured NO₂ concentration in the control standard deviates significantly from the actual concentration, a correction factor will be applied to measured NO_x concentrations.

$$\text{Factor} = \frac{\text{Concentration (actual)}}{\text{Concentration (measured)}}$$

D. METHOD VALIDATION

Prior to analysis of test samples, the hydrazine/UDMH method will be validated as follows:

Two gas samples containing nominally 1.3 percent and 0.1 percent by volume A-50 vapor in nitrogen will be prepared in teflon-lined gas sampling bags. Known volumes of these samples will be pulled through separate bubblers containing 20 milliliters each of 2 percent HCl solution. The solutions will then be analyzed by the proposed method to determine accuracy, precision, and percent recovery.

E. REPORTING

Hardcopies of raw data (chromatograms, spectra, titrator printouts, calculations) and analysis results will be forwarded directly to the program manager.