

Development of a Laboratory-Scale Test for Evaluating the Decomposition Products Generated Inside an Intact Fuselage During a Simulated Postcrash Fuel Fire

Timothy R. Marker
Louise C. Speitel

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16. Abstract <p>This report summarizes the research effort undertaken by the Federal Aviation Administration to develop a laboratory-scale test method for evaluating the products of combustion inside an intact transport category fuselage during exposure to a simulated external fuel fire. An oil-fired burner, configured in accordance with Title 14 Code of Federal Regulations Part 25.856(b) Appendix F Part VII, was used to simulate the fuel fire, and a 4- by 4- by 4-foot steel cube box was used to mount representative test samples. The cube box simulated an intact fuselage and served as an enclosure to collect emitted gases during fire exposure. Test samples representing a variety of fuselage constructions were evaluated, including a noncontemporary prototype structural composite material (without thermal acoustic insulation). A typical cross section consists of a 40- by 40-inch aluminum panel representing the fuselage skin and the accompanying thermal acoustic insulation blanket behind the skin. Two thermal acoustical configurations were also tested. The first contained a heat-stabilized polyacrylonitrile fiber blanket. The second contained a ceramic paper barrier sandwiched under a fiberglass blanket. Each was encased by a thin metallized polyvinylfluoride moisture barrier. These burnthrough-resistant configurations were primarily run to provide a baseline for comparing the emitted gas concentrations with that of the prototype structural composite material.</p> <p>A specialized Fourier Transform Infrared/total hydrocarbon gas analysis system was used to continually measure the products of combustion collected within the enclosure. Additional analyzers continuously measured the amount of carbon monoxide, carbon dioxide, and oxygen in the collected stream.</p> <p>During the testing, it was determined that a prototype multi-ply structural composite material produced minimal quantities of toxic and flammable gases during a 5-minute fire exposure. Approximately 7 plies of the 13-ply composite material were delaminated by the fire exposure. By comparison, the aluminum skin/insulation configurations generated higher gas concentrations.</p> <p>Subsequent full-scale testing of these material systems will provide gas scaling factors. The goal is to use this laboratory-scale test and scaling factors to predict decomposition products for an aircraft postcrash fuel fire.</p>					
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LIST OF SYMBOLS AND ACRONYMS

CH ₄	Methane
C ₂ H ₂	Acetylene
C ₂ H ₄	Ethylene
C ₂ H ₆	Ethane
CH ₂ CHCHO	Acrolein
C ₃ H ₈	Propane
C ₆ H ₆	Benzene
C ₆ H ₅ NH ₂	Aniline
C ₆ H ₅ OH	Phenol
CO	Carbon monoxide
COCl ₂	Carbonyl chloride
COF ₂	Carbonyl fluoride
CO ₂	Carbon dioxide
COS	Carbonyl sulfide
H ₂ O	Water
HBr	Hydrogen bromide
HCl	Hydrogen chloride
HCN	Hydrogen cyanide
HF	Hydrogen fluoride
NH ₃	Ammonia
NO	Nitrogen oxide
N ₂ O	Nitrous oxide
NO ₂	Nitrogen dioxide
O ₂	Oxygen
SO ₂	Sulfur dioxide
ZnSe	Zinc selenide
ACM	Advanced composite material
CFR	Code of Federal Regulations
CLS	Classical least squares
FAA	Federal Aviation Administration
FTIR	Fourier Transform Infrared
LEL	Lower explosive limit
NDIR	Nondispersive infrared analyzer
PAN	Polyacrylonitrile
PVF	Polyvinylfluoride
THC	Total hydrocarbon

EXECUTIVE SUMMARY

A laboratory-scale test was developed to evaluate the thermal decomposition gases that could possibly be generated inside an intact fuselage during a postcrash fuel fire. The test consisted of an oil-fired burner configured in accordance with Title 14 Code of Federal Regulations Part 25.856(b) Appendix F Part VII to simulate the fuel fire, and a 4- by 4- by 4-foot steel cube box used to mount representative test samples. The cube box simulated an intact fuselage and served as an enclosure to collect emitted gases during fire exposure. Test samples representing several fuselage constructions were evaluated. One of the samples was a prototype epoxy/graphite structural composite that was representative of the fuselage construction in a next-generation, all-composite transport aircraft. Two fuselage configurations were initially evaluated consisting of an aluminum skin panel and accompanying insulation materials that met the new burnthrough standard: a ceramic-based lightweight barrier in conjunction with standard fiberglass batting and a heat-stabilized polyacrylonitrile fiber. Each was encased by a thin metallized polyvinylfluoride moisture barrier. These traditional configurations were primarily run to provide a baseline for comparing the emitted gas concentrations with those of the prototype structural composite material.

A process Fourier Transform Infrared analyzer was used to continuously measure the toxic and flammable gases collected within the enclosure. Additional analyzers measured the concentration of total hydrocarbons as propane, carbon monoxide, carbon dioxide, and oxygen. During the testing, it was determined that the prototype structural composite material produced minimal quantities of toxic and flammable gases during a 5-minute fire exposure.

Approximately 7 plies of the 13-ply composite panel were damaged by the fire. In contrast, the aluminum skin/insulation configurations generated higher gas concentrations than the composite materials during a 5-minute fire exposure.

Subsequent full-scale testing of these material systems will provide a scaling factor for predicting full-scale test decomposition products.

INTRODUCTION

PURPOSE.

This report describes the research effort undertaken by the Federal Aviation Administration (FAA) to develop a laboratory-scale test method for measuring decomposition products inside an intact transport category fuselage during exposure to an adjacent external fuel fire. The test results from this laboratory-scale test and future full-scale burnthrough tests of these materials will provide scaling factors.

BACKGROUND.

In a majority of survivable accidents accompanied by fire, ignition of the interior of the aircraft is caused by burning jet fuel external to the aircraft as a result of fuel tank damage during impact. One important factor to occupant survivability is the integrity of the fuselage during an accident. Usually, there are two possibilities that exist in a survivable aircraft accident: (1) an intact fuselage and (2) a compromised fuselage from either a crash rupture or an opened emergency exit, which allows direct impingement of external fuel fire flames on the cabin materials. Based on a consideration of past accidents, experimental studies, and fuselage design, it is apparent that the fuselage rupture or opening represents the worst-case condition and provides the most significant opportunity for fire to enter the cabin [1]. Past FAA regulatory actions governing interior material flammability were based on full-scale tests employing a fuel fire adjacent to a fuselage opening in an otherwise intact fuselage. This scenario, in which the cabin materials were directly exposed to the intense thermal radiation emitted by the fuel fire, represented a severe but survivable fire condition and was used to develop improved material flammability test standards. However, in some crash accidents, the fuselage remained completely intact (no rupture or openings) and fire penetration into the passenger cabin was the result of a burnthrough of the fuselage shell [2].

There are typically three barriers that a fuel fire must penetrate in order to burnthrough to the cabin interior: aluminum skin, thermal acoustical insulation, and the interior sidewall and floor panel combinations. The burnthrough resistance of aluminum skin is well known, lasting between 30 to 60 seconds, depending on the thickness. Thermal acoustical insulation, typically comprised of fiberglass batting encased in a thin moisture barrier film, can offer an additional 1 to 2 minutes protection if the material is not physically dislodged from the fuselage structure [3 and 4].

To evaluate potential improvements in burnthrough protection under realistic conditions, the FAA William J. Hughes Technical Center developed a full-scale fuselage burnthrough testing rig. The construction of this apparatus was the most practical approach for repetitive testing and systematic evaluations of singular components. A 20-foot-long steel cylindrical test rig was fabricated, and the test rig was then inserted between two halves of a Boeing 707 fuselage (figure 1). This test rig has a 12- by 8-foot section of the outer skin removed and can be mocked up with aluminum skin, thermal acoustical insulation, floor and sidewall panels, carpet, and cargo liner. The mocked up test rig extends beyond the 10-foot-long fire pan, eliminating problems that might occur if the edges of the installed fuselage materials were in direct exposure to the fuel

fire. Measurements of temperature, smoke, carbon monoxide (CO), carbon dioxide (CO₂), and oxygen (O₂) were taken inside the test rig along with video coverage at several locations to determine exact burnthrough locations and times.

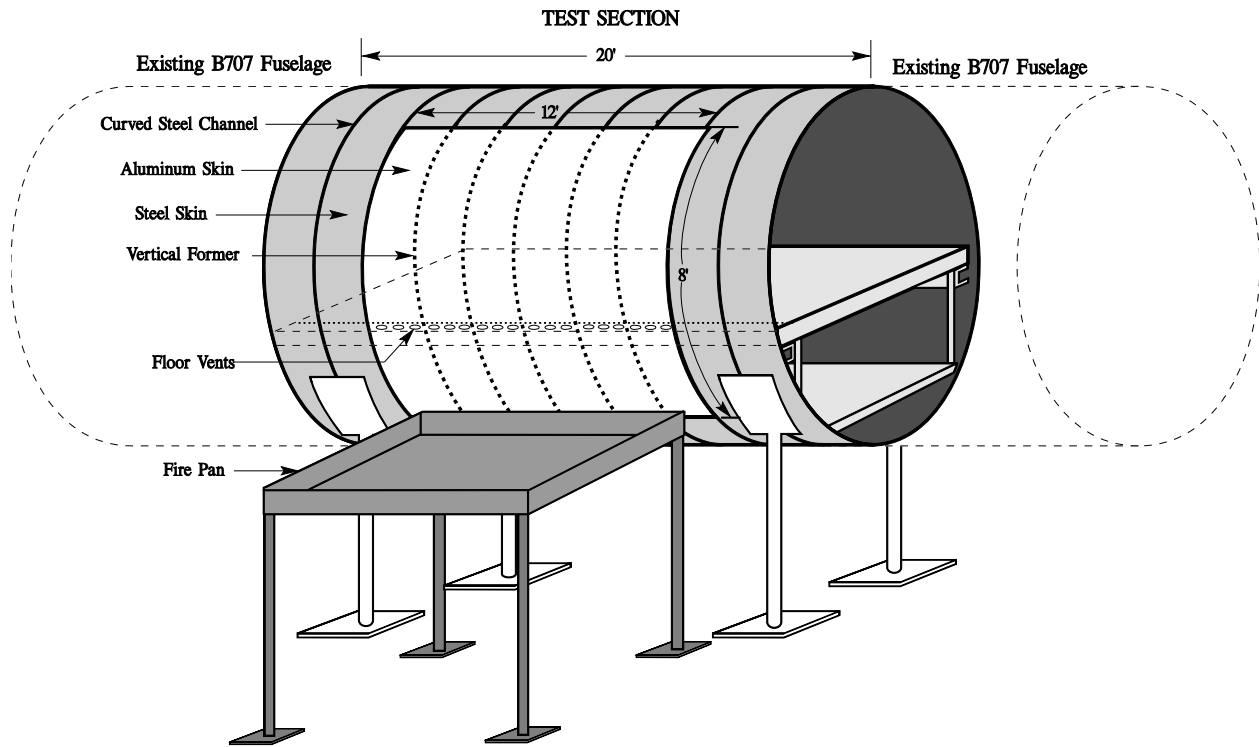


Figure 1. Full-Scale Fuselage Burnthrough Test Rig

Testing in this full-scale apparatus confirmed the enhanced burnthrough protection from a variety of materials including a thin, dot-printed, ceramic-based barrier that could be inserted into the existing fiberglass insulation bags. A heat-stabilized, oxidized polyacrylonitrile (PAN) fiber was equally capable at preventing a fully developed fuel fire from entering the cabin for as much as 8 minutes. When compared to current insulation materials, which were shown to fail in as little as 2 minutes, effective fire barriers offer increased life-saving potential during a postcrash fire accident in which the fuselage remains intact [5].

Based on the encouraging findings of the full-scale tests, which showed the potential benefit of increased burnthrough protection, the FAA began work on a laboratory-scale test that could replicate the full-scale conditions. The new laboratory test would evaluate the burnthrough protection capabilities of materials without the expense of running a full-scale test. The initial test apparatus used an oil-fired burner, similar to that used in other FAA flammability tests, along with a steel box used to mount a mock-up, 24- by 24-inch test sample consisting of the outer aluminum skin and thermal acoustic insulation beneath it. The box also allowed combustion product monitoring of its contents and the viewing of the unexposed side of the sample using a video camera (figure 2).

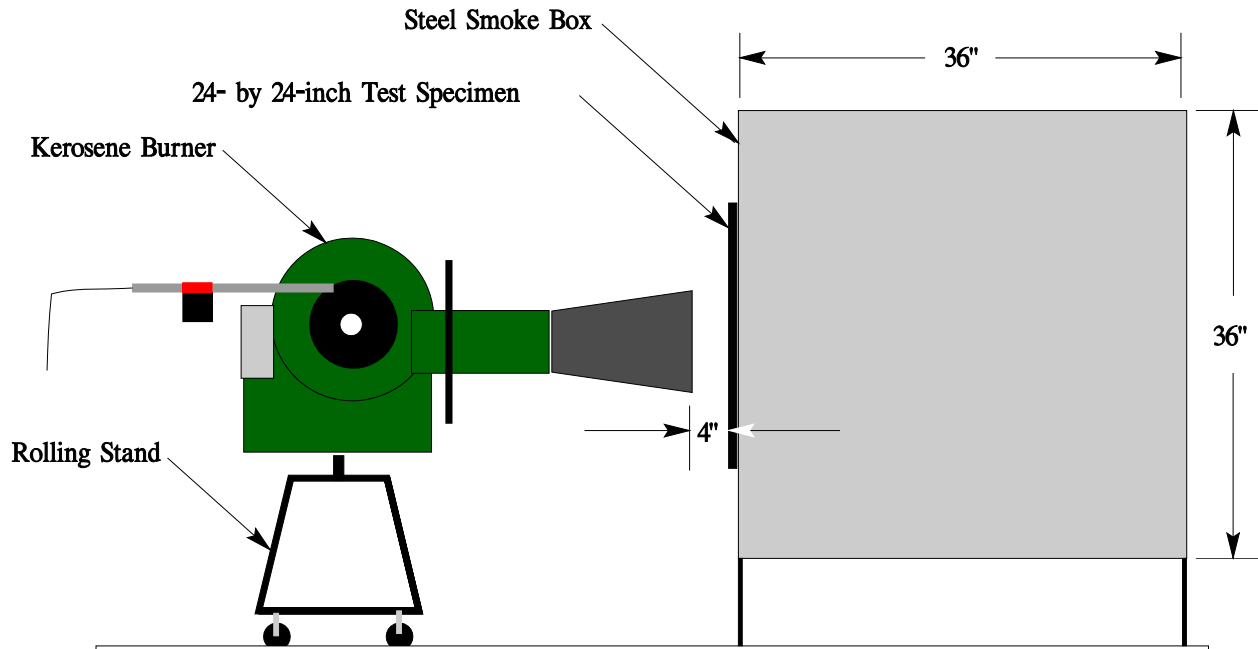


Figure 2. Initial Laboratory-Scale Burnthrough Test Rig

The box portion of the apparatus was eventually abandoned and replaced with a grid-style frame to mount insulation samples. Eventually, the aluminum skin sample on the exterior of the apparatus was also abandoned, as this became a cumbersome, time-consuming task. Since aluminum skin offers little practical opportunity for fire hardening, the focus of extending the burnthrough resistance has been on the thermal acoustical insulation and, to a much lesser extent, the floor/sidewall panel combination and related components. Full-scale fire tests have shown that appreciable gains in burnthrough resistance can be achieved by either protecting or replacing the current fiberglass thermal acoustical insulation.

In the streamlined test, samples of thermal acoustic insulation were mounted to the sample holder frame (figure 3), which resembled the former and stringer structure in transport aircraft, and exposed to the oil-fired burner flame for a period of 4 minutes (figure 4). The finalized test exposure condition consisted of a flame temperature of 1900°F and heat flux of 16.0 Btu/ft²sec. The burner output cone was situated 4 inches from the outer plane of the sample holder frame at an angle of 30° with respect to horizontal. This configuration yielded results that correlated with previous full-scale tests that used identical materials. During the testing, it was also determined that the method of attaching the insulation blankets to the test sample structure had a critical impact on the effectiveness of the insulation material.

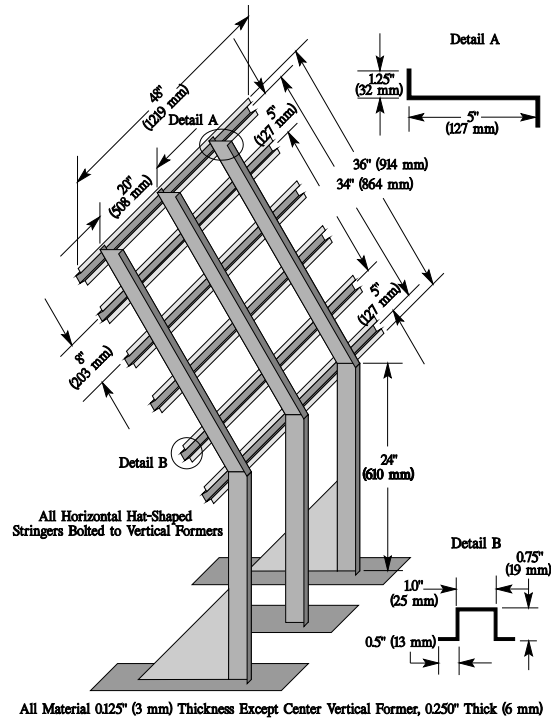


Figure 3. Insulation Burnthrough Test Sample Holder

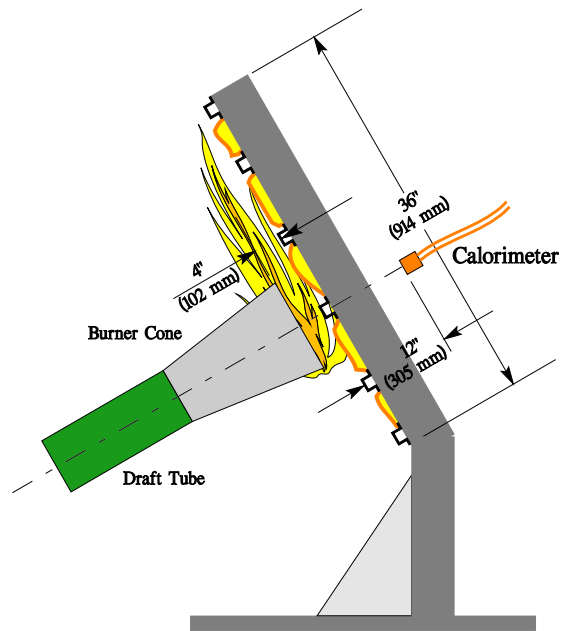


Figure 4. Finalized Insulation Burnthrough Test Apparatus

To evaluate and improve the reproducibility of the finalized test apparatus worldwide, a number of round-robin test series were conducted. During a typical round robin, several different types of insulation blanket test samples were identically prepared, shipped to participating laboratories, and tested. Test results were tabulated, compared, and analyzed to determine the degree of fluctuation or scatter of data from the laboratories. The standard deviation of test results from four round-robin tests had shown that the data scatter had been reduced during each test series, indicating that the test was reproducible.

In September 2003, the FAA implemented a new regulation for the flammability of thermal acoustic insulation. The new regulation consisted of two new flammability test standards: one measured the ability to prevent an in-flight fire and the other to resist postcrash fire flame penetration or burnthrough [6]. The new burnthrough requirement affected all primary thermal acoustic insulation in the lower half of the fuselage and specified resistance to flame penetration for 4 minutes. The burnthrough test standard is required in Title 14 Code of Federal Regulations (CFR) Part 25.856(b) and is described in Appendix F Part VII (herein referred to as Appendix F Part VII).

TOXICITY.

Although full-scale testing confirmed the enhanced burnthrough protection from a variety of materials, there were concerns over the potential toxicity of the PAN material during thermal decomposition because acrylonitriles generate hydrogen cyanide (HCN) when exposed to fire. Therefore, when the PAN material was tested in the full-scale rig, HCN, CO, and CO₂ were measured at two locations within the cabin, one close to the burnthrough area and another near the front of the test fuselage, both at a height of 5 feet 6 inches. Although the concentrations of HCN and CO were relatively high, it was determined that their levels were nonlethal and nonincapacitating during a 5-minute exposure, which is the maximum time required to evacuate from an analysis of past accidents (figure 5).

Although it was apparent that a robust, burnthrough-resistant insulation system would result in a much more survivable cabin atmosphere than a traditional, non-burnthrough-resistant system, the buildup of any toxic gases resulting from the decomposition of the insulation material was a concern. For this reason, an additional laboratory test was developed to evaluate the decomposition products that could possibly be generated inside an intact fuselage during a postcrash fuel fire.

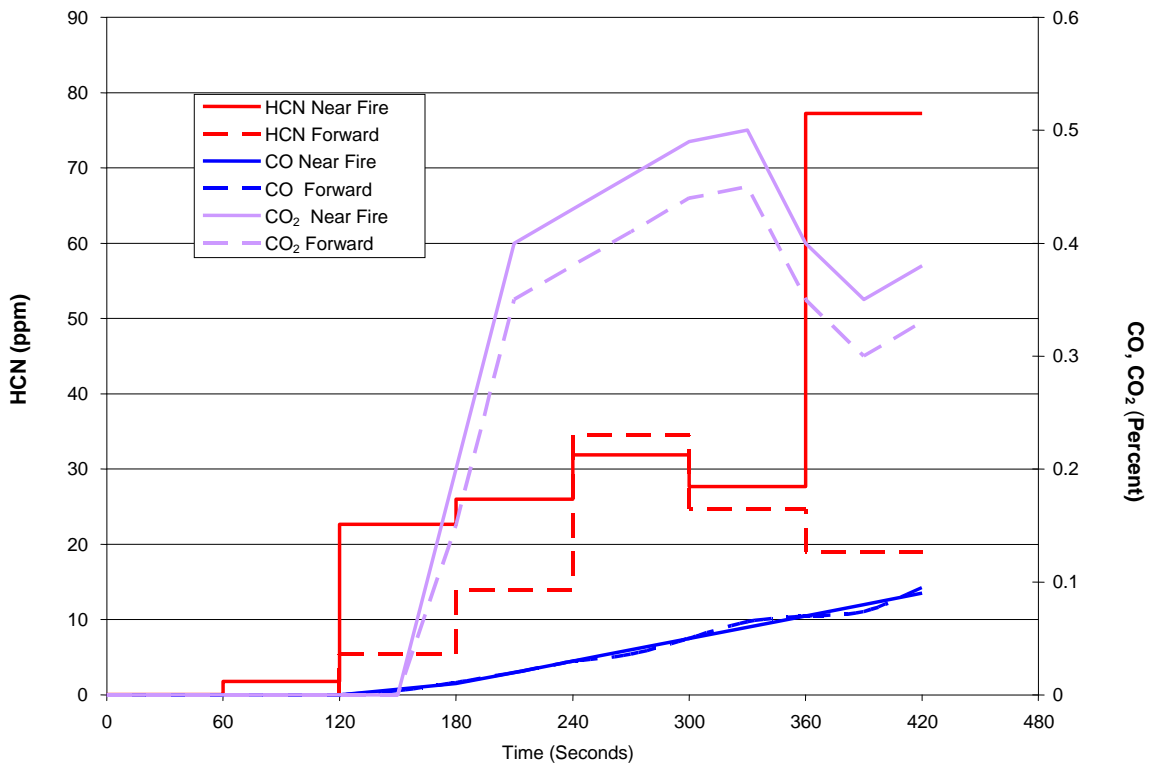


Figure 5. Gases Measured During Full-Scale Test With Polyacrylonitrile Insulation

EXPERIMENTAL

DEVELOPMENT OF A LABORATORY-SCALE TESTING RIG FOR EVALUATING TOXICITY.

To evaluate the decomposition products that could possibly be generated inside an intact fuselage during a postcrash fuel fire, a test was devised that incorporated the burner apparatus used in the insulation burnthrough test. The burner equipment was configured in accordance with Appendix F Part VII, which requires a 1900°F flame and a heat flux of 16 Btu/ft²sec. To capture all combustion products given off during exposure of a representative test sample to the fire, an enclosure was needed. The use of a large, 4- by 4- by 4-foot steel cube box was the most practical method. Due to its relatively large size, the top corner of the box was flattened to allow for clearance under the test area fume hood. A large opening measuring 40 by 40 inches was used on the exposed face of the box to accommodate a test sample, and the face was positioned 4 inches from the burner cone. The cube box simulated an intact fuselage and served as an enclosure to collect gas emissions during fire exposure (figure 6). The area/volume ratio was greater than what might be expected in a survivable crash in order to concentrate the gas levels and facilitate gas analysis.

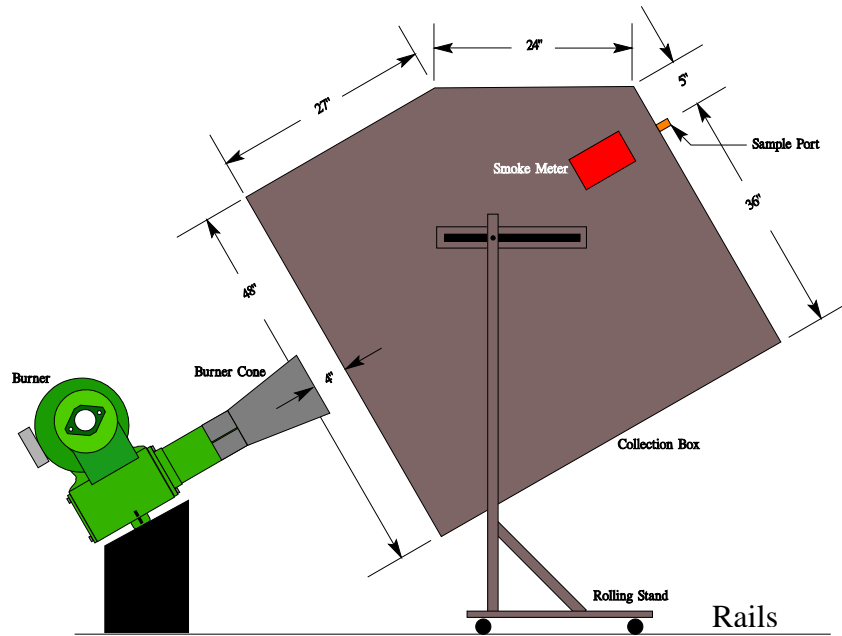


Figure 6. Test Apparatus for Evaluating the Toxicity of Insulation Materials

The insulation sample was mounted to the enclosure in a manner that prevented the intrusion of combustion products from the test burner flame. To accomplish this, the face of the steel box enclosure was recessed around the periphery of the opening, which allowed for a 40- by 40-inch test sample to be flush-mounted. A steel flange/gasket was mounted on top of the insulation sample and bolted into place around the perimeter using stud-mounted 1/4-inch bolts (figure 7) to seal the sample edges.

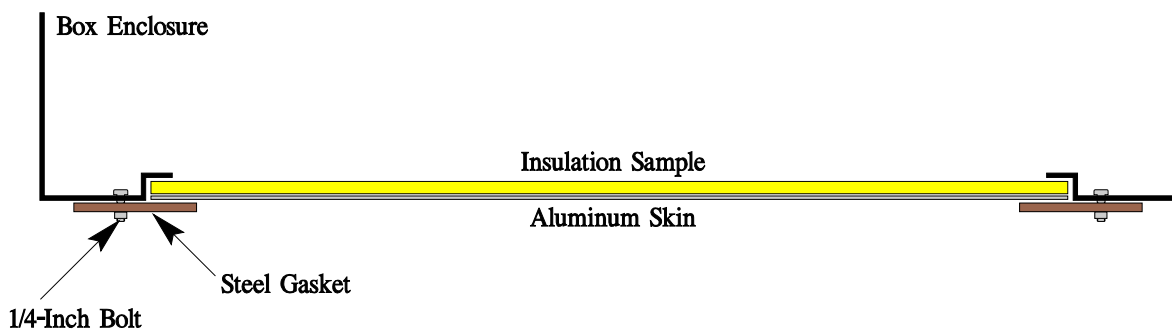


Figure 7. Box Enclosure Mounting System

During a typical test, the insulation sample and aluminum skin is mounted onto the box enclosure, and all bolts are securely tightened. The entire box enclosure is removed from the immediate burner area. The burner is then lit and warmed up for 2 minutes to ensure steady-state conditions. Following this warm-up period, the entire box is quickly rolled into position in front of the burner and subjected to the flame for 5 minutes. A rail and stop system ensure correct positions of the box. An in-box thermocouple can detect early burnthrough, and the test can be terminated to prevent damage to the gas-sampling equipment. All gas-sampling lines are flexible

and are supported from above so they will not interfere with the quick movement of the rolling box both into and away from the fire.

Under this test configuration, the thermal decomposition products in the enclosure were continuously monitored 5 inches below the top of the enclosure. A specialized extractive Fourier Transform Infrared (FTIR)/total hydrocarbon (THC) process analysis system was used for the analysis of the toxic gases CO, CO₂, carbonyl chloride (COCl₂), carbonyl fluoride (COF₂), acrolein (CH₂CHCHO), HCN, nitrogen oxide (NO), nitrous oxide (N₂O), nitrogen dioxide (NO₂), ammonia (NH₃), sulfur dioxide (SO₂), carbonyl sulfide (COS), benzene (C₆H₆), aniline (C₆H₅NH₂), phenol (C₆H₅OH), hydrogen fluoride (HF), hydrogen chloride (HCl), and hydrogen bromide (HBr), as well as water (H₂O) and the hydrocarbons methane (CH₄), acetylene (C₂H₂), ethylene (C₂H₄), ethane (C₂H₆), propane (C₃H₈), and THC as propane. This analysis system was designed to minimize errors found in conventional FTIR systems employed for combustion gas analysis [7]. The selected gases include the primary decomposition products of advanced composite material (ACM) and PAN materials identified in previous studies [8 and 9].

Additional continuous analyzers measured the concentration of CO, CO₂, and O₂ in the sample stream. The sampling line leading to the THC and FTIR analyzers was heated to minimize condensation of sample gases.

METHODS OF ANALYSIS.

GAS ANALYZERS. THCs were monitored using a Rosemont Model NGA2000 total hydrocarbon analyzer, type MLT-2R. This is a nondispersive infrared analyzer (NDIR). A Rosemount OM-11ea polarographic analyzer was used to monitor oxygen. CO and CO₂ were monitored using Rosemount 880a CO and CO₂ NDIR analyzers. These analyzers use Luft-type detectors filled with CO and CO₂, respectively. Additional optical filters enhanced selectivity. The Luft detector operates on the principle of common absorbance with the detector gas. The gas cells have a path length of approximately 3 mm for the CO and CO₂ analyzers. This short path length allows quantification at the strongest CO and CO₂ absorbance bands, resulting in greater accuracy for complex combustion gas mixtures. The CO and CO₂ analyzers were used in this study to provide confidence in the FTIR method development for high concentrations of these gases.

THE FTIR ANALYZER.

The FTIR Spectrometer and Data Acquisition. A Midac Model I2001F FTIR Spectrometer¹ with a 4-meter cell was used for all tests. The optical path of this cell is over 1000 times longer than the 3-mm CO and CO₂ NDIR analyzer cells, contributing to a much greater sensitivity and lower limit of detection. The sample cell is nickel, the mirrors are coated with gold, and the windows are zinc selenide (ZnSe). The cell volume is approximately 160 ml. The interferometer, beamsplitter, and windows are constructed of ZnSe with a germanium coating for moisture protection. The detector is a liquid nitrogen-cooled Mercury Cadmium

¹ Certain commercial equipment, instruments, or materials are identified in this technical note to specify the experimental procedure. Such identification does not imply that the material or equipment is the best available for the purpose or endorsement by the FAA.

Telluride detector, providing more than an order of magnitude additional sensitivity. All interface optics are gold-coated for high light throughput and corrosion resistance. The sample cell has a horizontal orientation to minimize buildup of soot on the cell mirrors.

All tests were conducted in an extractive mode at 0.5 cm^{-1} resolution, with an average of 16 scans every 9 seconds. All calibration spectra were obtained at 0.5 cm^{-1} and were prepared for the FAA by the FTIR manufacturer at 170°C for all gases except H_2O , CH_2CHCHO , $\text{C}_6\text{H}_5\text{NH}_2$, $\text{C}_6\text{H}_5\text{OH}$, C_6H_6 , NH_3 , N_2O , C_3H_8 , and COS . The 170°C H_2O calibration spectra were prepared in-house at the FAA William J. Hughes Technical Center using a syringe calibration system, (which was built into the sampling system). The flow rate through the cell was 1-2 liters/min [7]. The NH_3 , C_6H_6 , and N_2O spectra were obtained at 25°C , and the COS and C_3H_8 spectra were obtained at 121°C from the Midac Spectral Library. Aniline, acrolein, and phenol spectra were obtained at 100°C and a 0.5-cm^{-1} resolution from the Environmental Protection Agency Standards Library.

Cell pressure and temperature are monitored and recorded with each spectrum. The FTIR software performs Beer's Law calculations for each analyte in each test spectra to correct the analyte concentration for any pressure and temperature variations from the calibration spectra.

Data were collected, analyzed, and plotted using the following commercial software: Midac Autoquant Pro software, Operant LLC Essential FTIR, and Microsoft Excel[®]. Spectral bands for 24 gases were selected to have minimal common absorbance. Another requirement for spectral band selection is that it must have a low absorbance, so that the calibration is linear over a wide concentration range. Broad nonoverlapping bands were selected when possible.

The FTIR Method. The method identifies the 24 gases to be analyzed and the spectral regions for applying the classical least squares (CLS) method within the $650\text{- to }4500\text{-cm}^{-1}$ spectral range. Figure 8 illustrates the calibration spectra and spectral regions used in this FTIR method. Only the selected regions highlighted in figures 8-18 were used in the CLS method. For each test spectrum and analyte gas, the method subtracts spectral regions that are common with spectral regions of other gases in the method. A few very wide regions were selected for the water spectra, ensuring that water interferences would be subtracted from sample spectra. Spectral regions were selected for each gas to minimize interferences with other gases.

Many regions were used for CO and CO_2 for the various calibration concentrations to enable quantification in low-absorbing regions that exhibit the best linearity. Absorbencies less than 0.1 generally provide acceptable linearity. Calibration spectra were selected for each gas to define each piecewise-linear calibration curve. The minimum number of spectra needed to obtain accurate calibration curves is selected.

Figure 10 shows that the slope of the selected CO_2 region (the shoulder of the highly absorbing, clipped, CO_2 peak) for the highest concentration CO_2 spectrum is large, resulting in a decreased accuracy at the higher CO_2 concentrations.

All calibration spectra and test spectra were obtained at a unit gain. A triangular apodization, a Mertz phase correction, and a resolution of 0.5 cm^{-1} were used for all calibration

and test spectra. The method creates a spreadsheet with the time profiles of the concentration and error (residuals) data for the 24 gases. Gas concentrations were reported as zero for each gas and spectra if the residuals for the spectral region were 50% or greater than the calculated concentration.

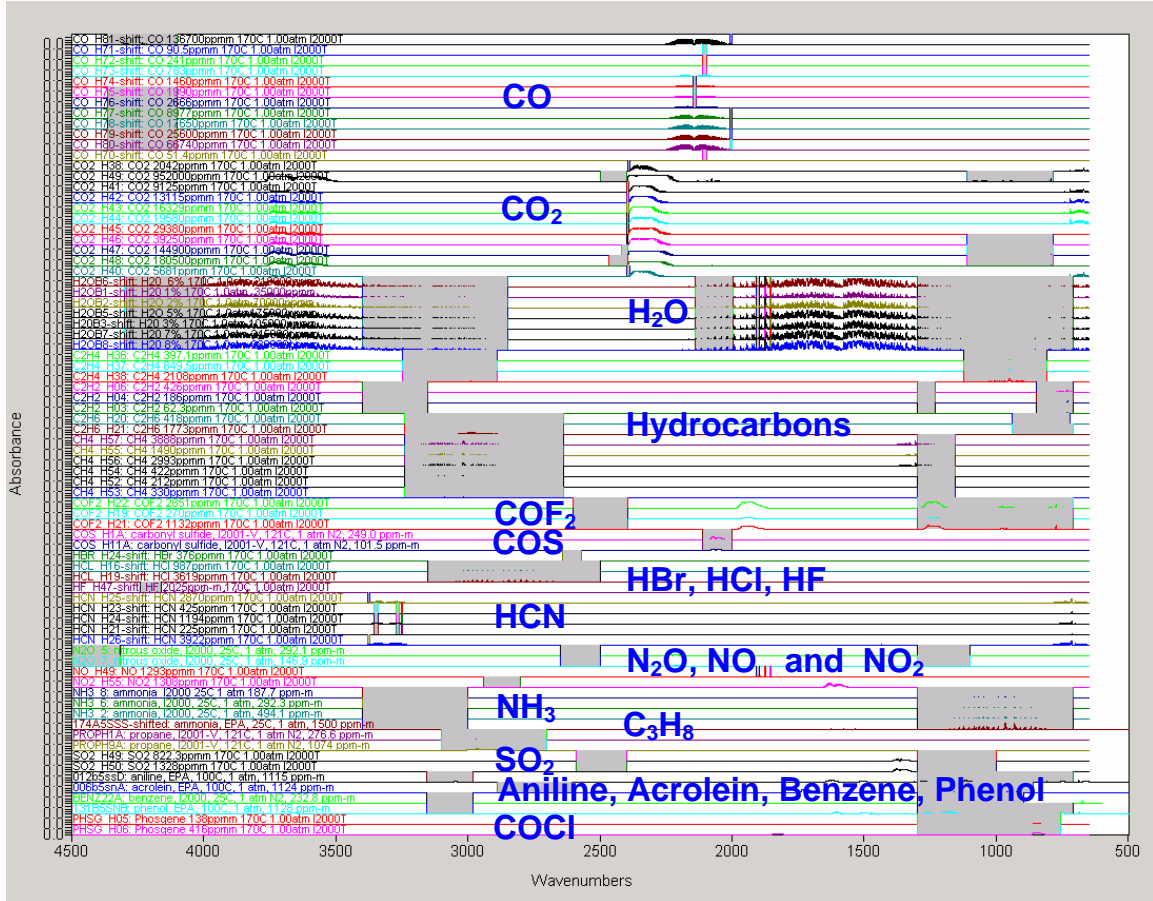


Figure 8. Calibration Spectra and Selected Regions for FTIR Analysis

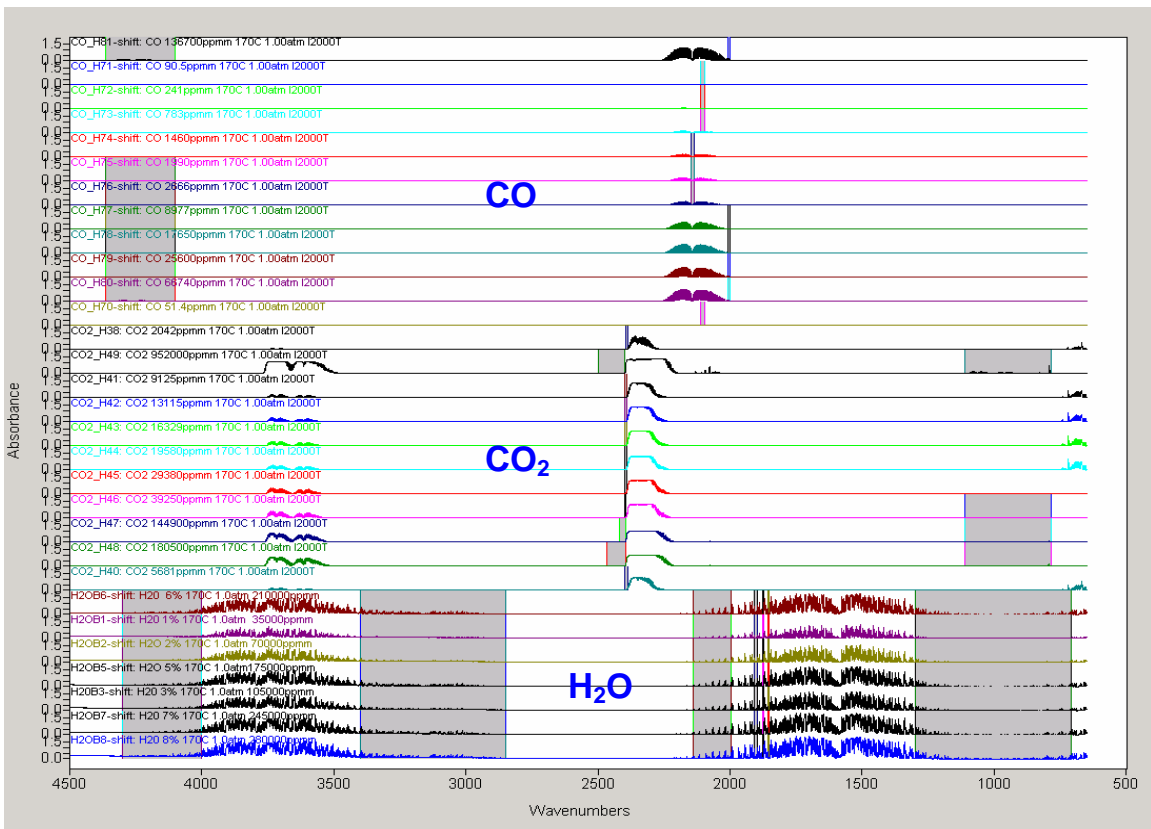


Figure 9. CO, CO₂, and H₂O Calibration Spectra and Regions

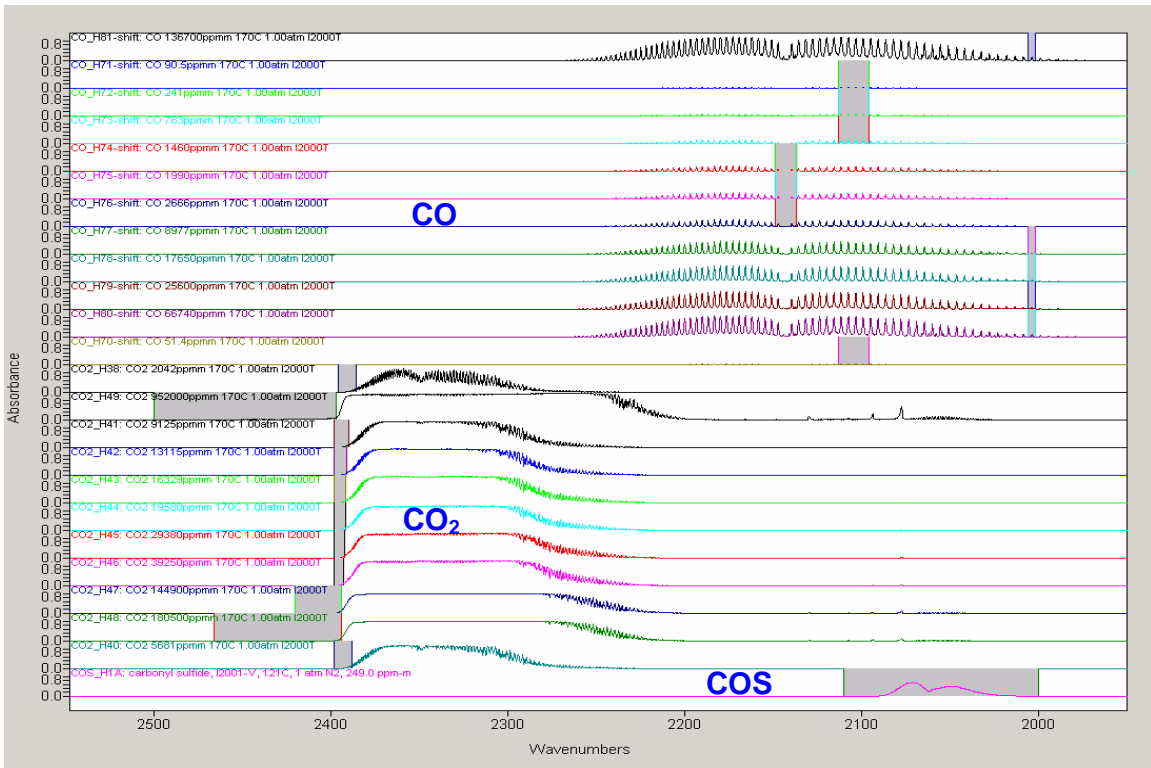


Figure 10. Expanded View of CO, CO₂, and COS Calibration Spectra and Regions

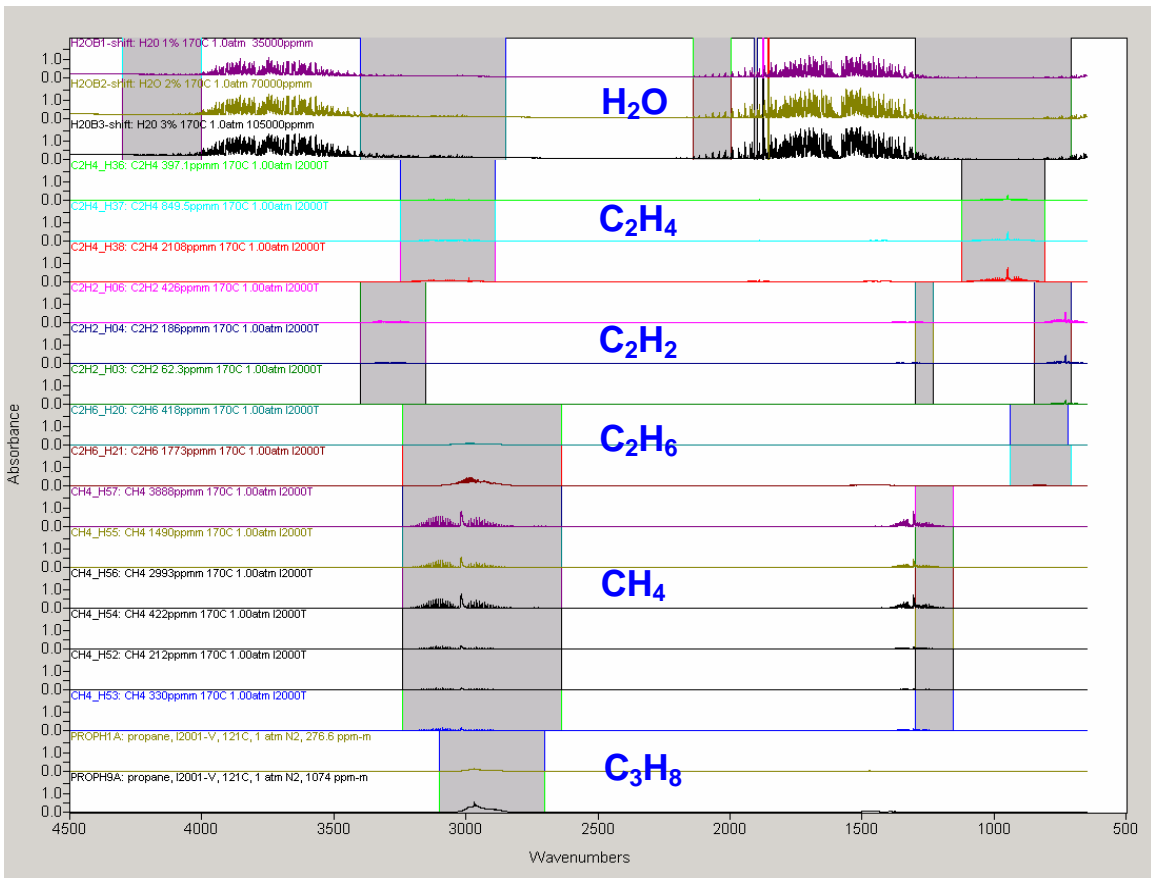


Figure 11. H₂O, C₂H₄, C₂H₂, C₂H₆, CH₄, and C₃H₈ Calibration Spectra and Regions

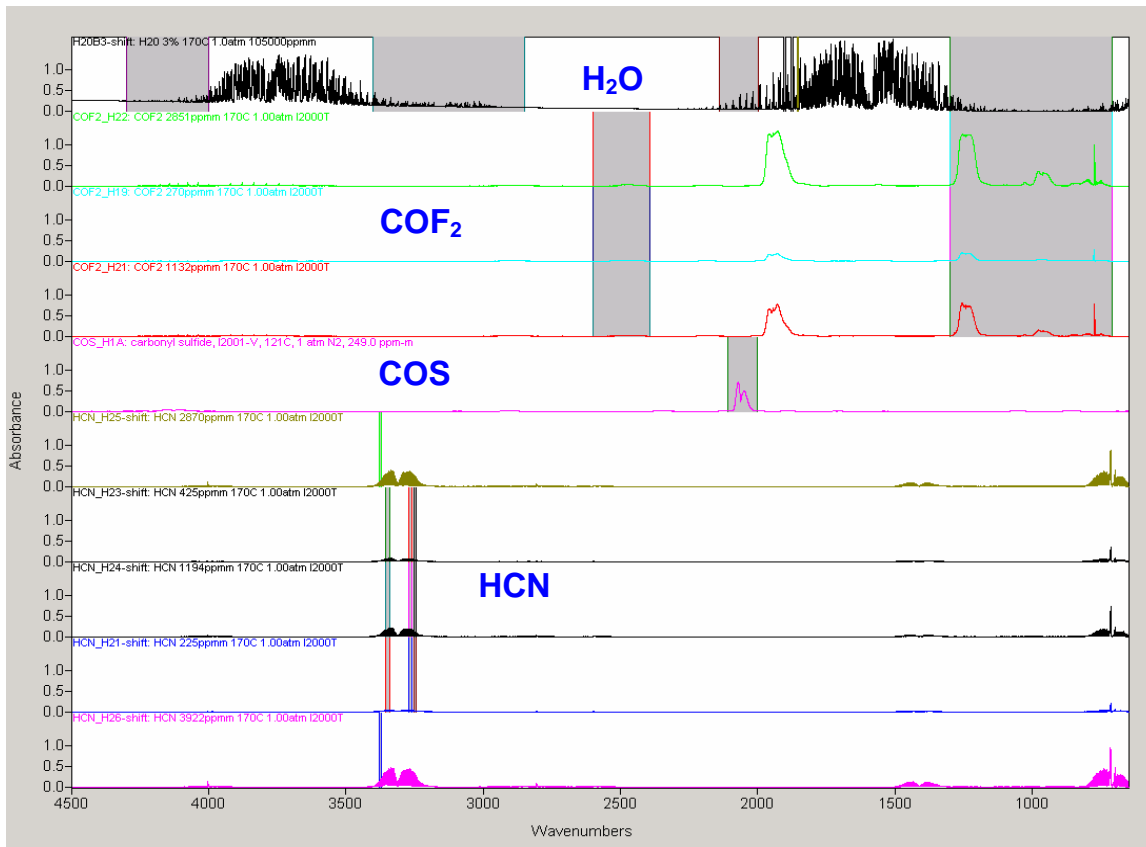


Figure 12. H₂O, COF₂, COS, and HCN Calibration Spectra and Regions

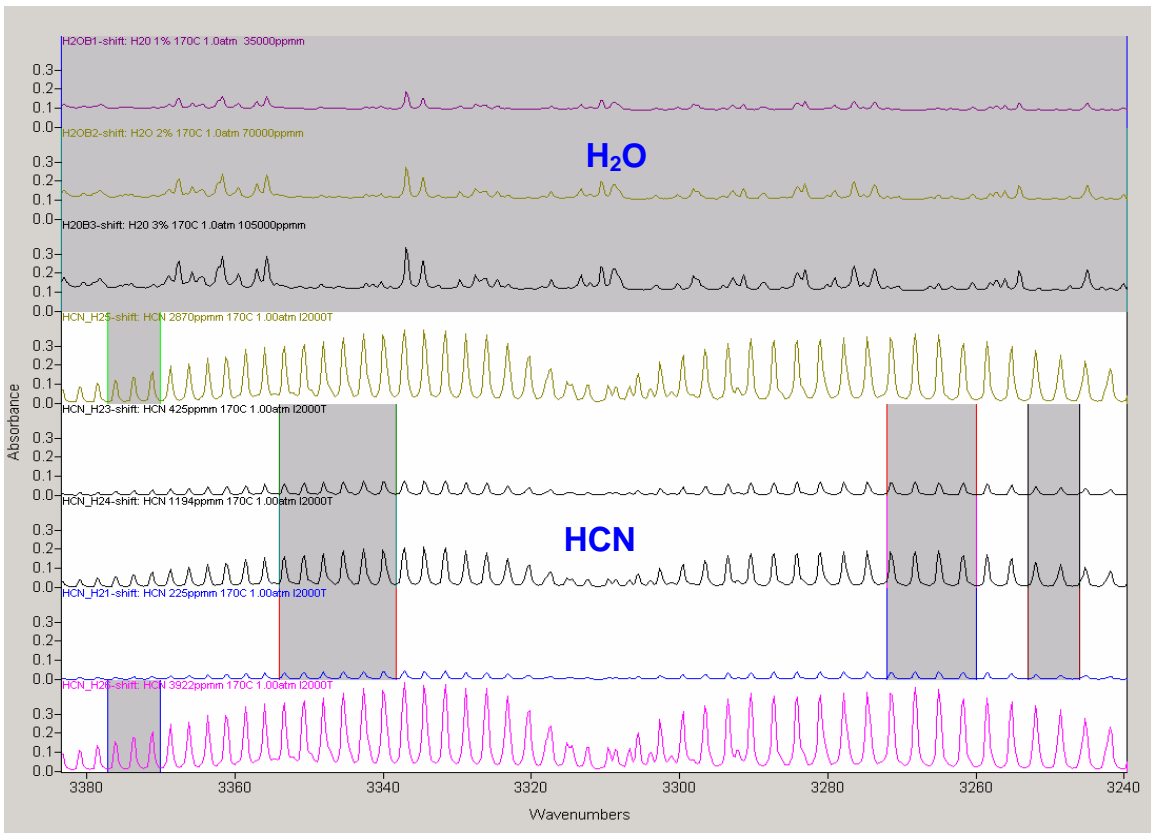


Figure 13. Expanded View of H₂O and HCN Calibration Spectra and Regions

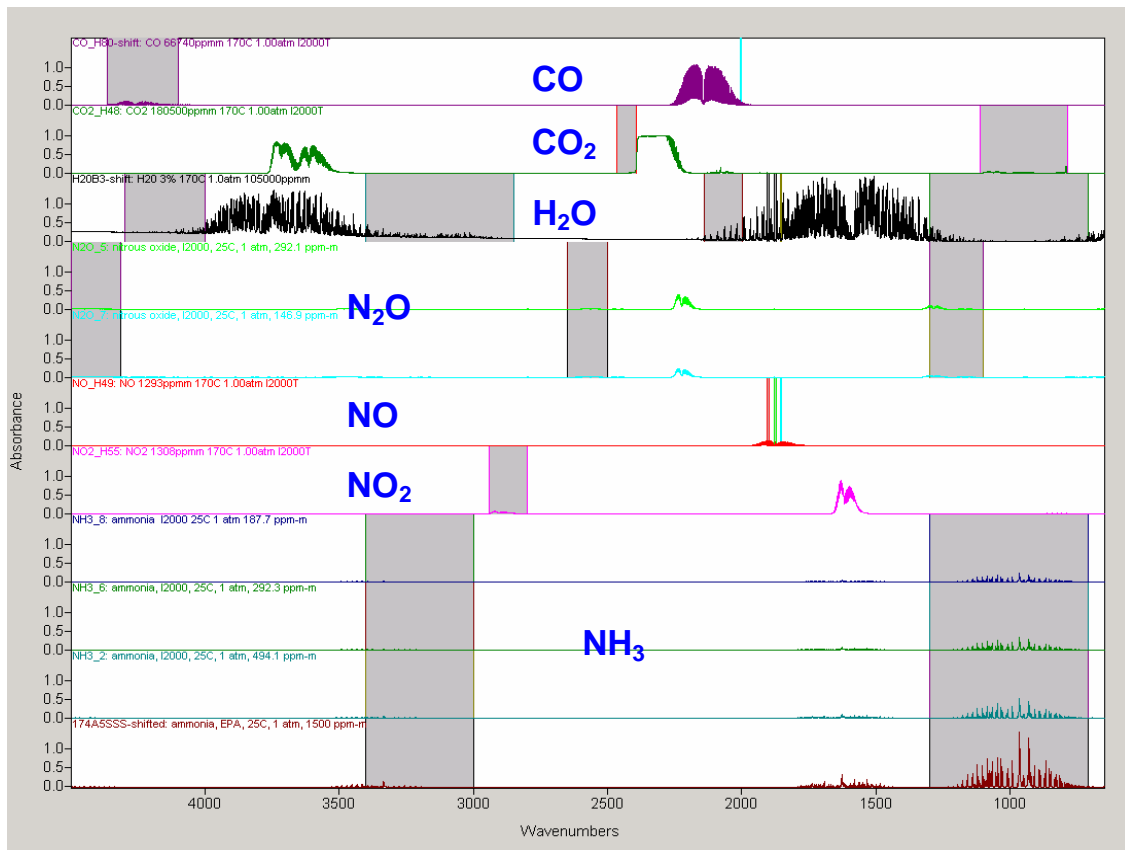


Figure 14. N₂O, NO, NO₂, and NH₃ Calibration Spectra and Regions

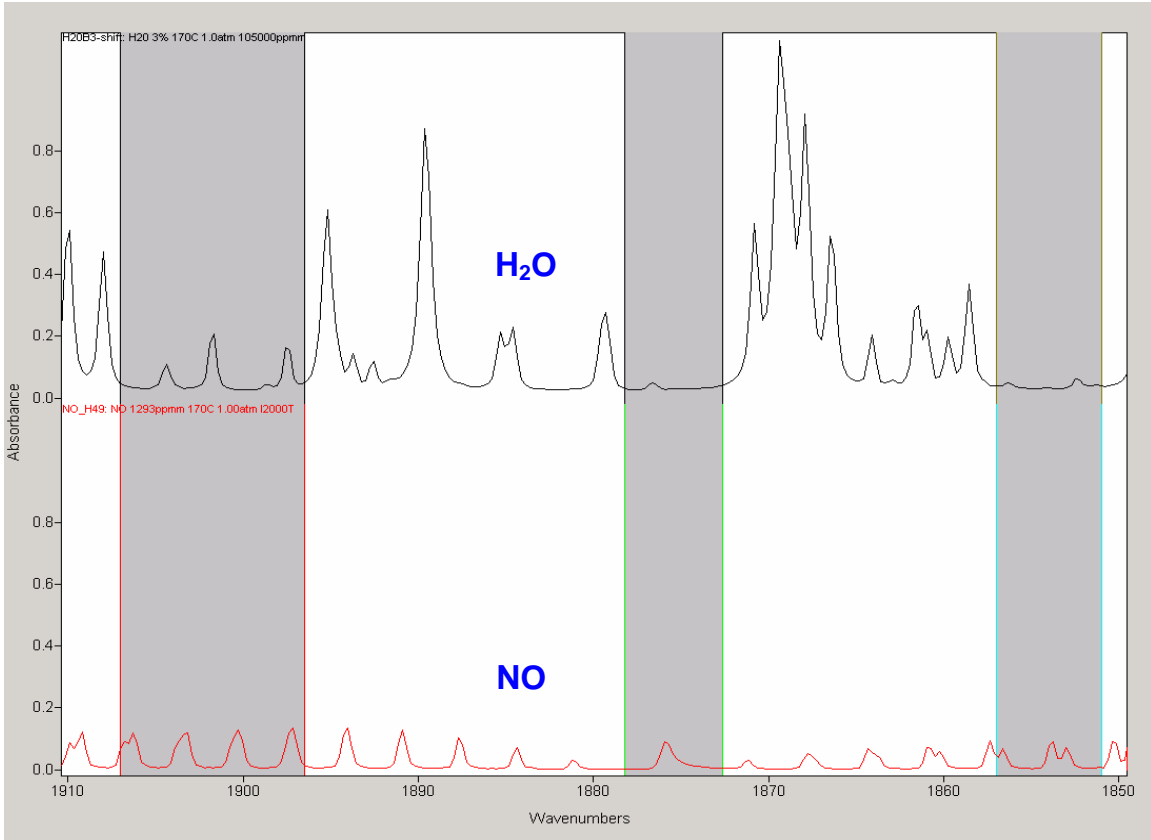


Figure 15. Expanded View of H₂O and NO Calibration Spectra and Regions

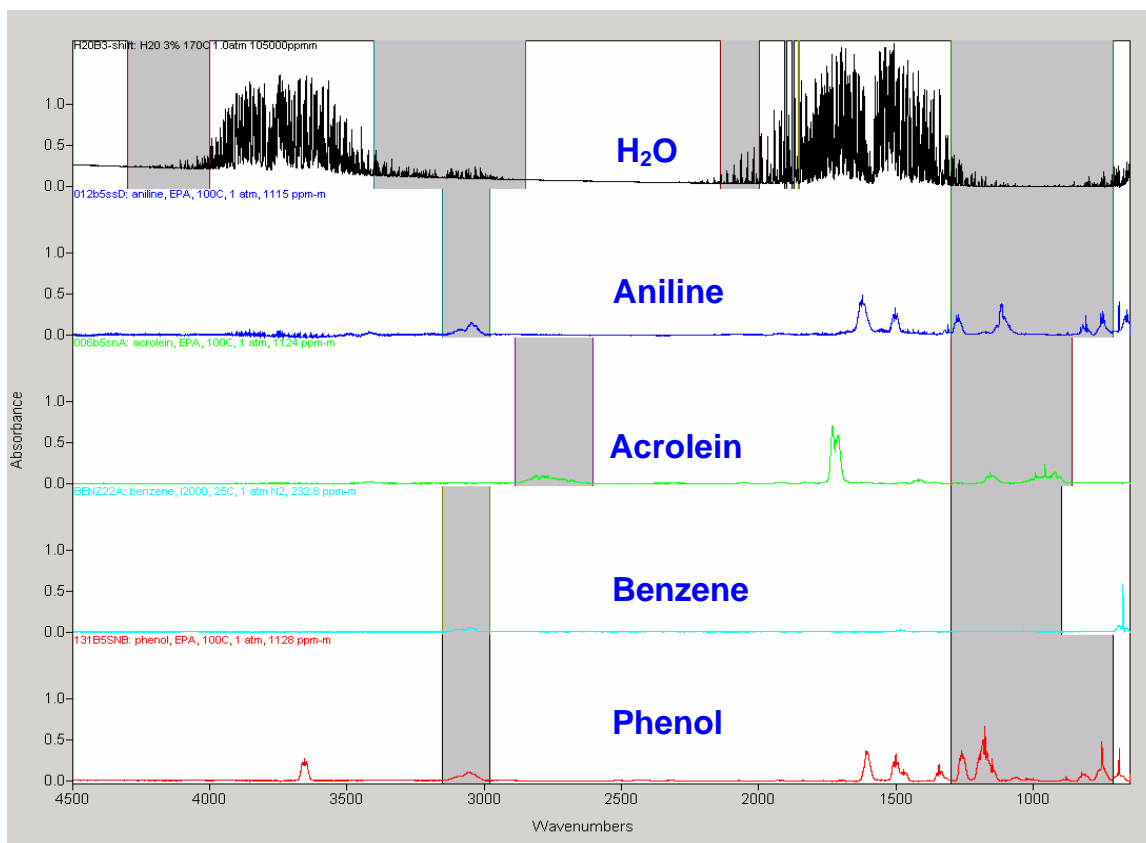


Figure 16. H₂O, Aniline, Acrolein, Benzene, and Phenol Calibration Spectra and Regions

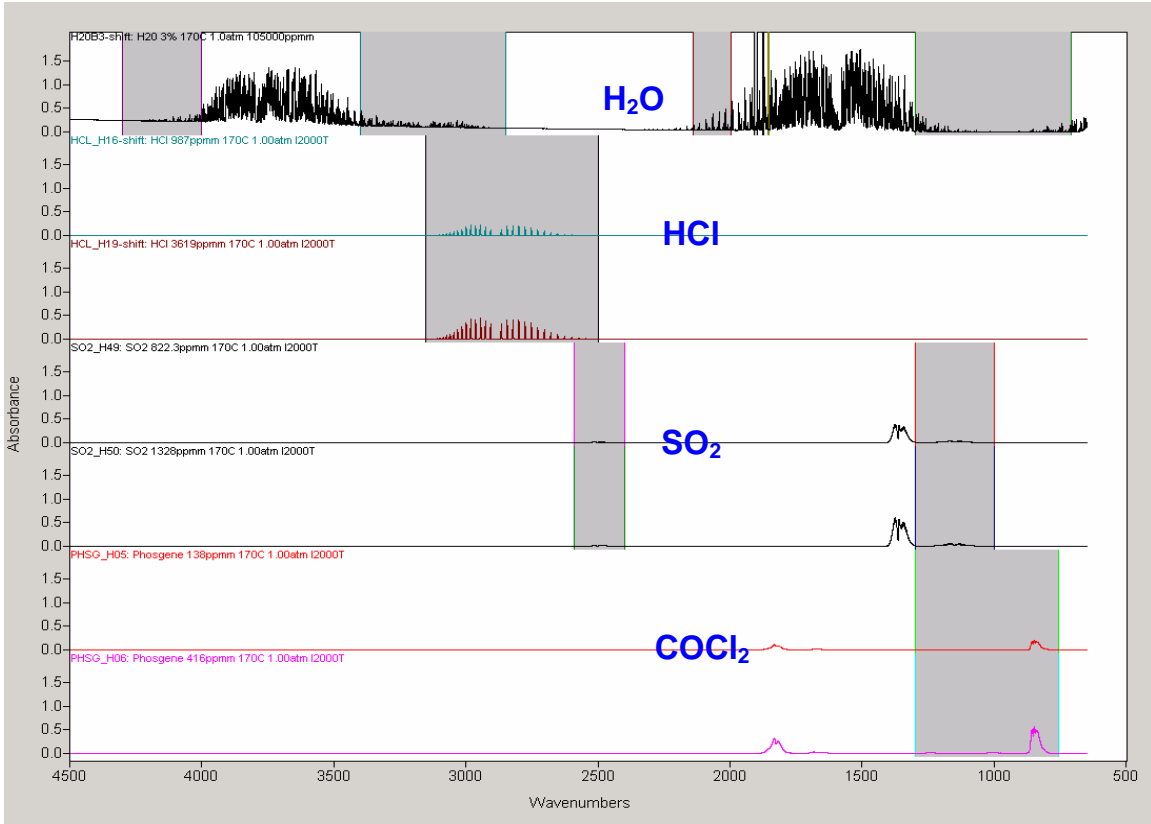


Figure 17. H₂O, HCl, SO₂, and COCl₂ Calibration Spectra and Regions

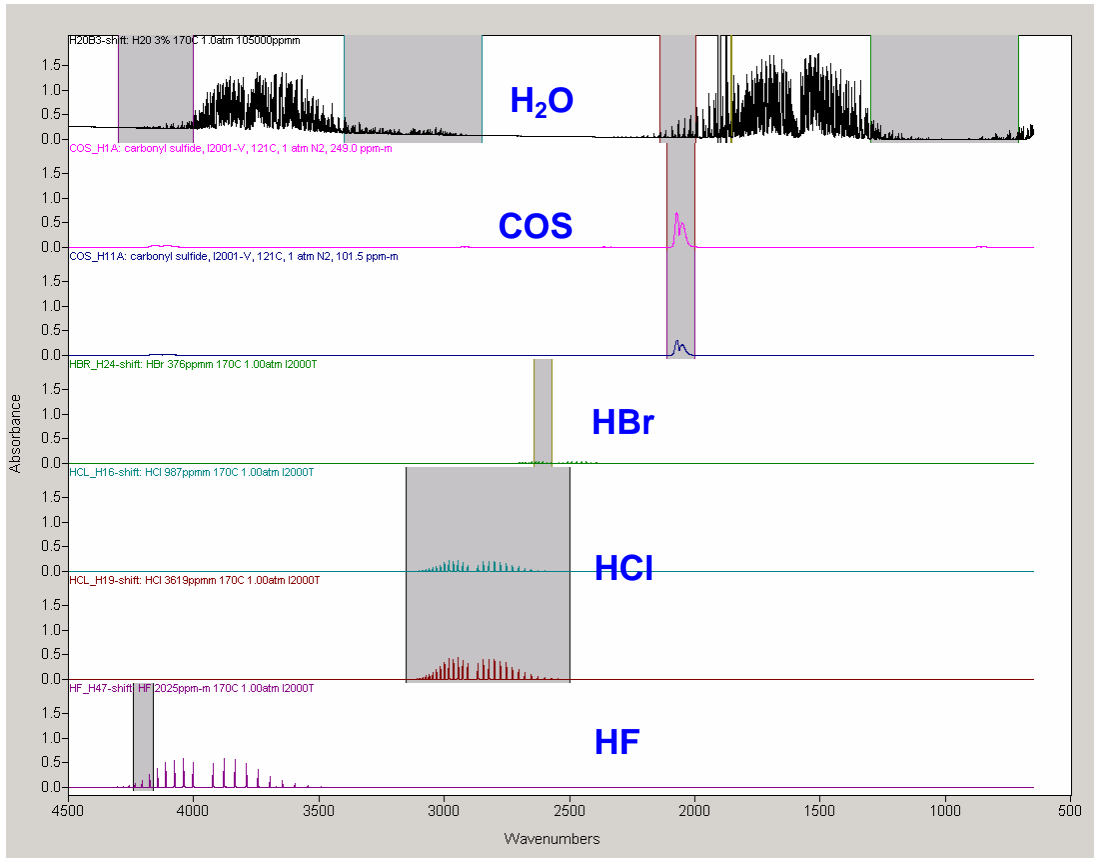


Figure 18. H₂O, COS, HBr, HCl, and HF Calibration Spectra and Regions

GAS-SAMPLING METHODOLOGY FOR THE FTIR AND THC ANALYZERS. Figure 19 is a schematic of the FTIR/THC sampling system. The entire path leading to the analyzers is heated to minimize condensation of analytes in the sampling system. The flexible, heated, Teflon[®]-lined, 20-ft (6.1-m) by 1/4-in. (0.64-cm) sample line runs from the test chamber to the sample conditioning filters that are housed in an oven. The heated sample line from the test chamber to the oven is composed of two 10-foot sections, each separately thermostated to 120°C. The entire sample path to the NDIR THC analyzer is thermostated to the maximum design temperature of that analyzer, 120°C. The line leading from the oven to the FTIR sample cell and the FTIR cell are thermostated to 170°C, the temperature at which the FTIR calibration standard library was developed. The separate thermostated circuits are indicated in figure 19.

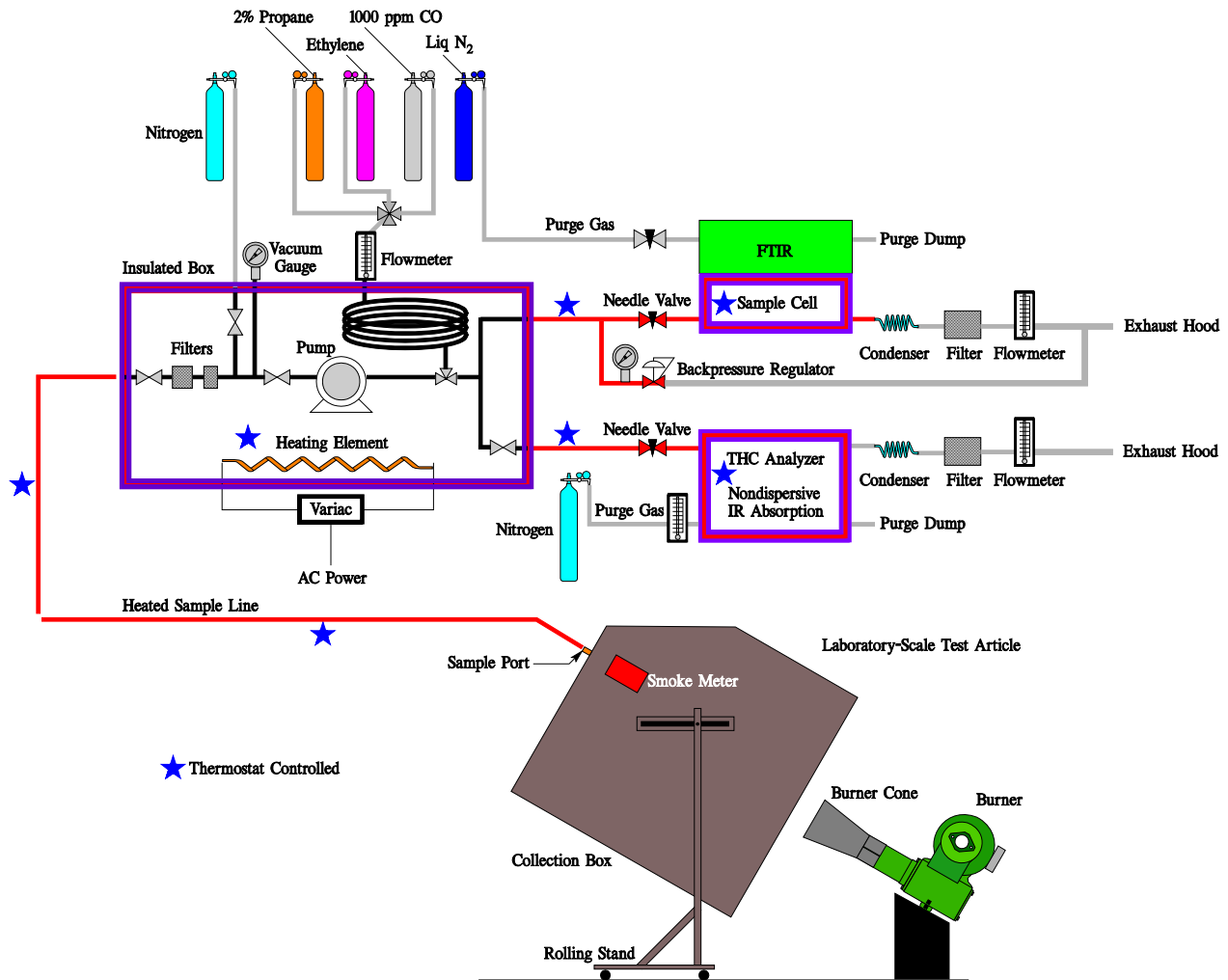


Figure 19. The FTIR and THC Sampling System

A three-way valve, downstream of the sample pump, can select either a calibration gas or a combustion gas input to the analyzers. The calibration gas flows from the gas cylinder through a heated, 13.1-ft (4-m) by 1/4-in. (0.64-cm) stainless steel coil. This coil enables preheating the calibration gas for analysis. The sample stream splits before it exits the oven to service the two analyzers. The tubing leading to the FTIR sample cell (and a point a few inches downstream of that cell) is maintained at 170°C, along with the sample cell. This preheats the sample stream to the FTIR calibration temperature of 170°C.

The gas sample is continuously drawn through the heated sample line, at a flow rate of approximately 11 liters per minute, and passes through a series of filters into the bellows pump. A high system flow rate (constant flow rate) through the 160-ml, 4-meter optical path length sample FTIR cell and the NDIR THC cell is used. This ensures a constant system response time throughout a fire test and from test to test, as particulates build up in the filters and the filter backpressure increases. The backpressure regulator output bypasses the analyzer. A needle valve at the inlet to each analyzer is set to provide a flow rate of 2.0 liters/min. A cooling coil of

1/4-inch (0.064-cm) copper tubing and a high-capacity filter protect the flow meter downstream of the sample cells.

A vacuum/pressure gauge monitors the filter for clogs and serves as a system check to monitor the filter housing for leaks after filter replacement. Valves can be opened to pressurize the filter with nitrogen and closed to check for leaks. Further details of the FTIR system can be found in reference 7.

TEST RESULTS AND DISCUSSION

INITIAL BASELINE TEST RESULTS (NO INSULATION SYSTEM—OPEN BOX).

To determine the type and amount of combustion products produced by the burner flame, an initial test was conducted without an insulation sample on the face of the steel cube box. A 1-foot length of 1/4-inch stainless steel tube extension was attached to the sample probe, and a thermocouple was teed to the connection with the heated sampling line. The sampling was terminated after less than 1 minute when the temperature exceeded 150°C to prevent the thermal decomposition of the Teflon sample line.

Figures 20 and 21 illustrate the gas concentration histories obtained for the open-box test. The high CO₂/CO ratio of about 300/1 and low hydrocarbon concentrations observed in this test (at 30 seconds) is characteristic of well-ventilated flaming combustion. Concentrations of H₂O and CO₂ exceeded 7%, and CO reached 248 ppm at 45 seconds into the test. HF, NO, SO₂, HCN, NH₃, C₆H₅NH₂, C₂H₆, CH₄, and C₃H₈ were observed. The HF may have condensed on the surface of the box during a previous fire test. Consideration must be given in interpreting the subsequent box test gas concentration profiles to the possible contribution of the fuel decomposition products to the gas yields.

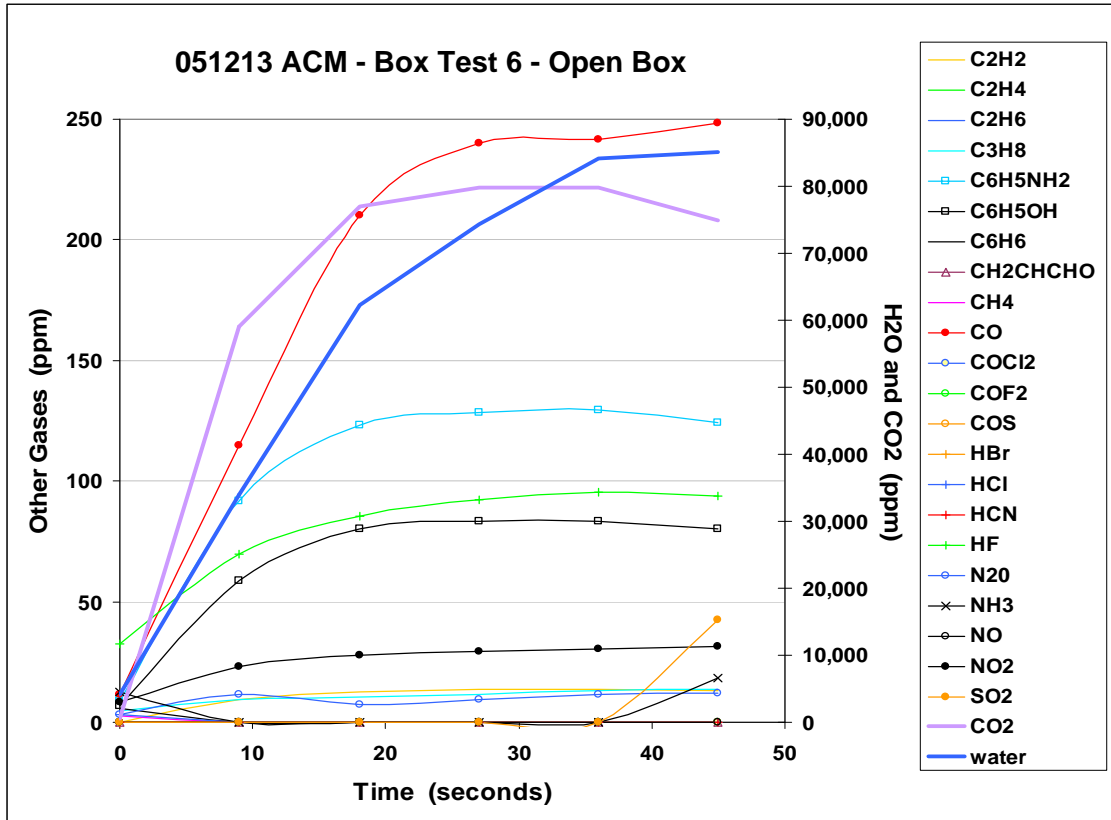


Figure 20. Concentration Histories for Open-Box Test Obtained by FTIR Analysis

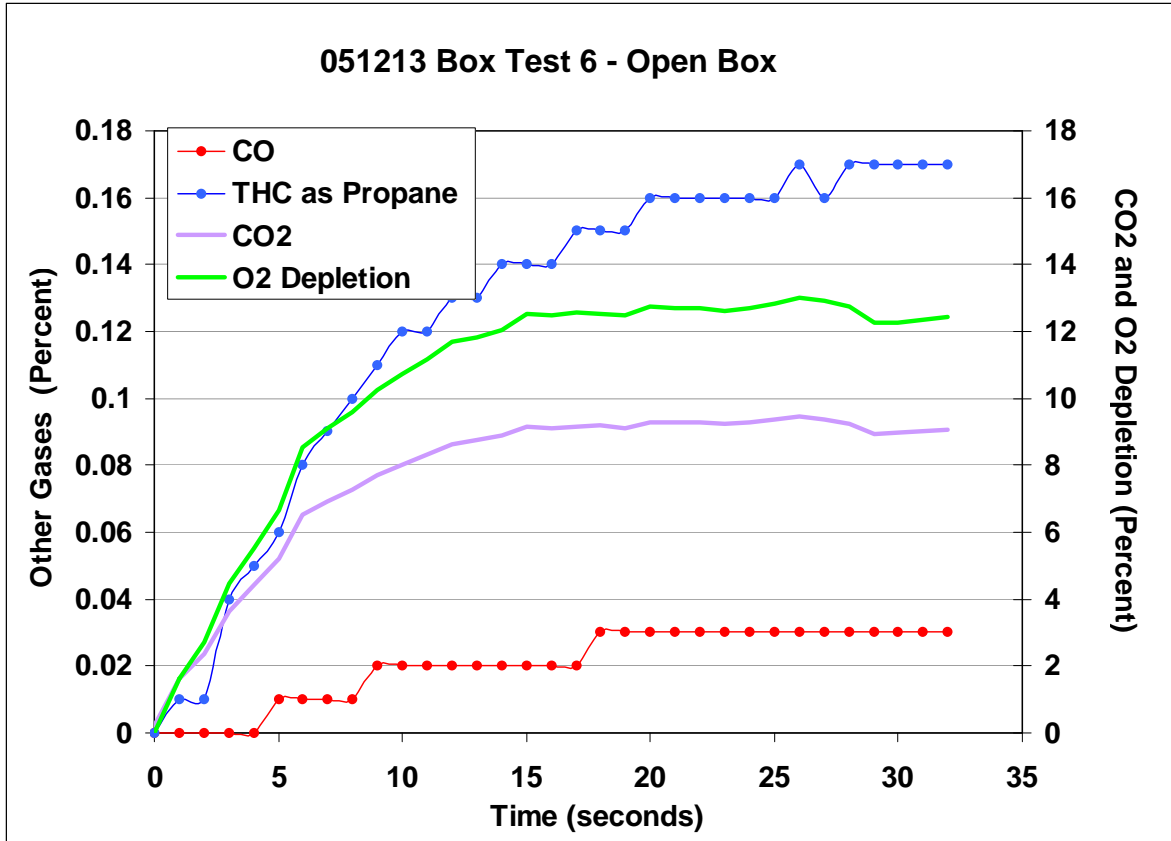


Figure 21. Concentration Histories for Open-Box Test Obtained by Gas Analyzers

LABORATORY-SCALE EVALUATION OF INSULATION MATERIALS MEETING THE NEW BURNTHROUGH STANDARD.

FUSELAGE CONSTRUCTIONS EVALUATED. The following material systems were tested as illustrated in figure 22.

- Aluminum skin panel with a ceramic paper barrier sandwiched under a fiberglass insulation blanket. The paper and blanket are encased by a thin metallized polyvinylfluoride (PVF) film.
- Aluminum skin panel with a heat-stabilized polyacrylonitrile fiber insulation blanket encased by a thin metallized PVF film
- A prototype epoxy/graphite structural composite material with no insulation blanket

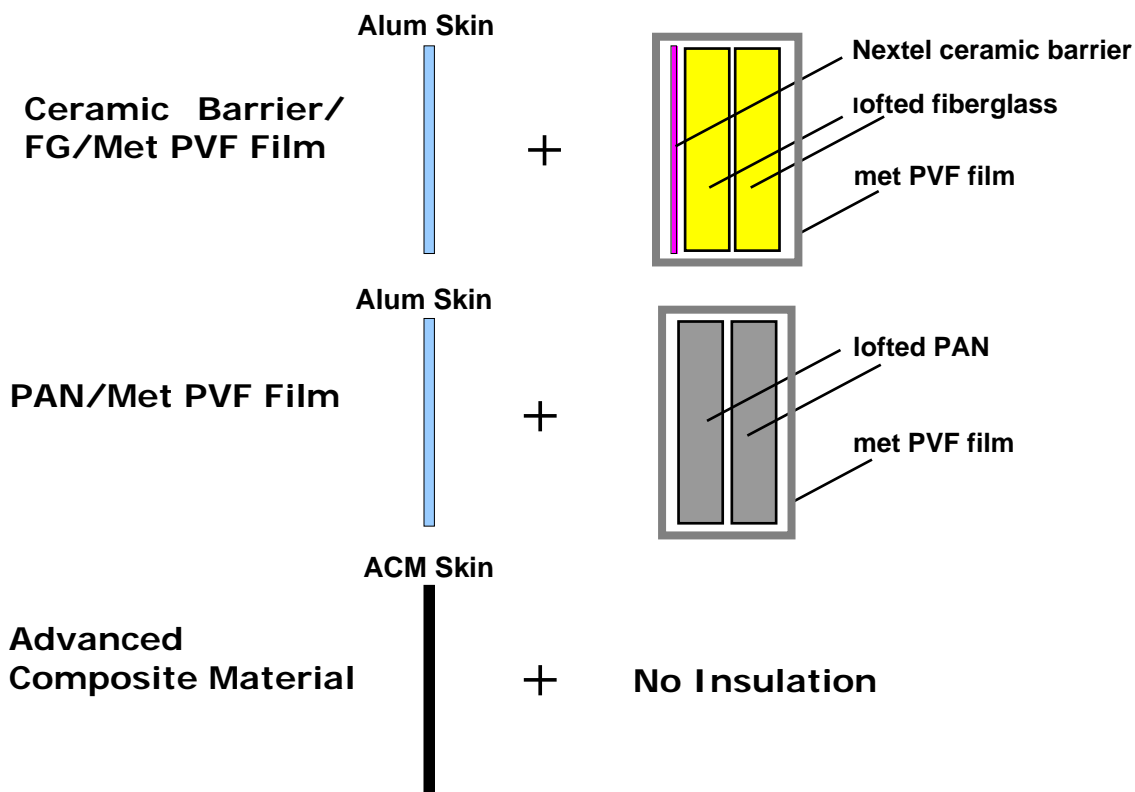
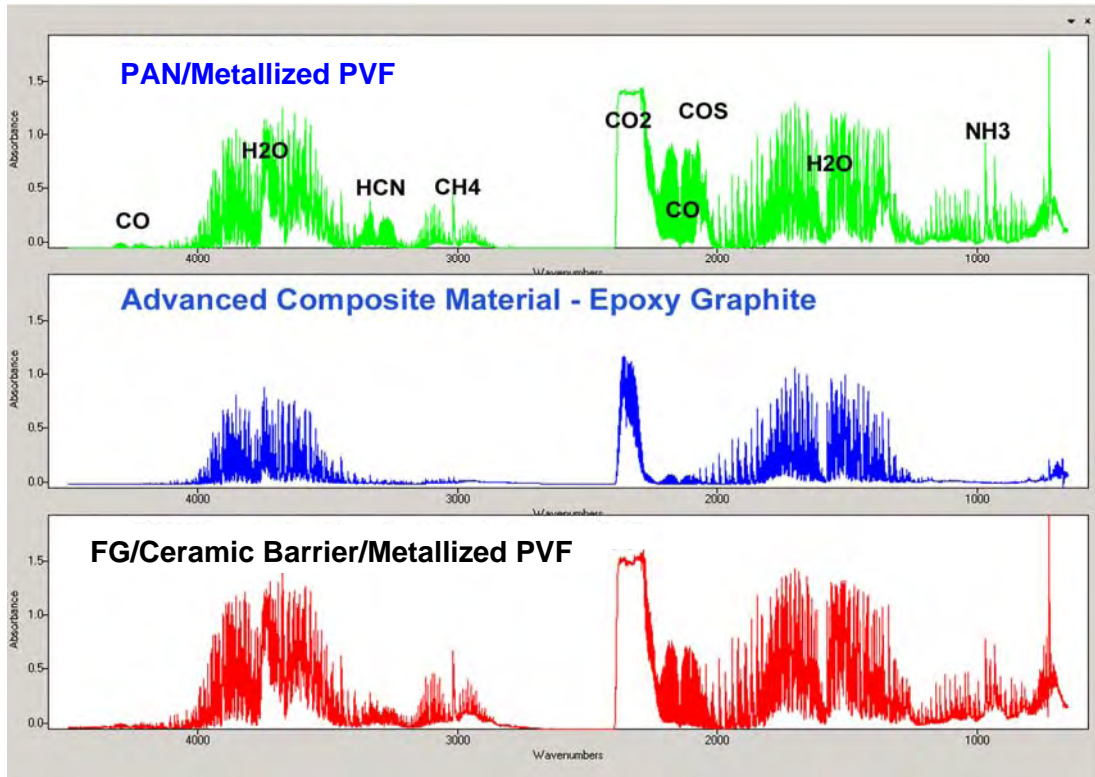


Figure 22. Material Systems Tested in Laboratory-Scale Apparatus

The FTIR spectra obtained 5 minutes into the test for the three material systems are shown in figure 23.



FG = Fiberglass

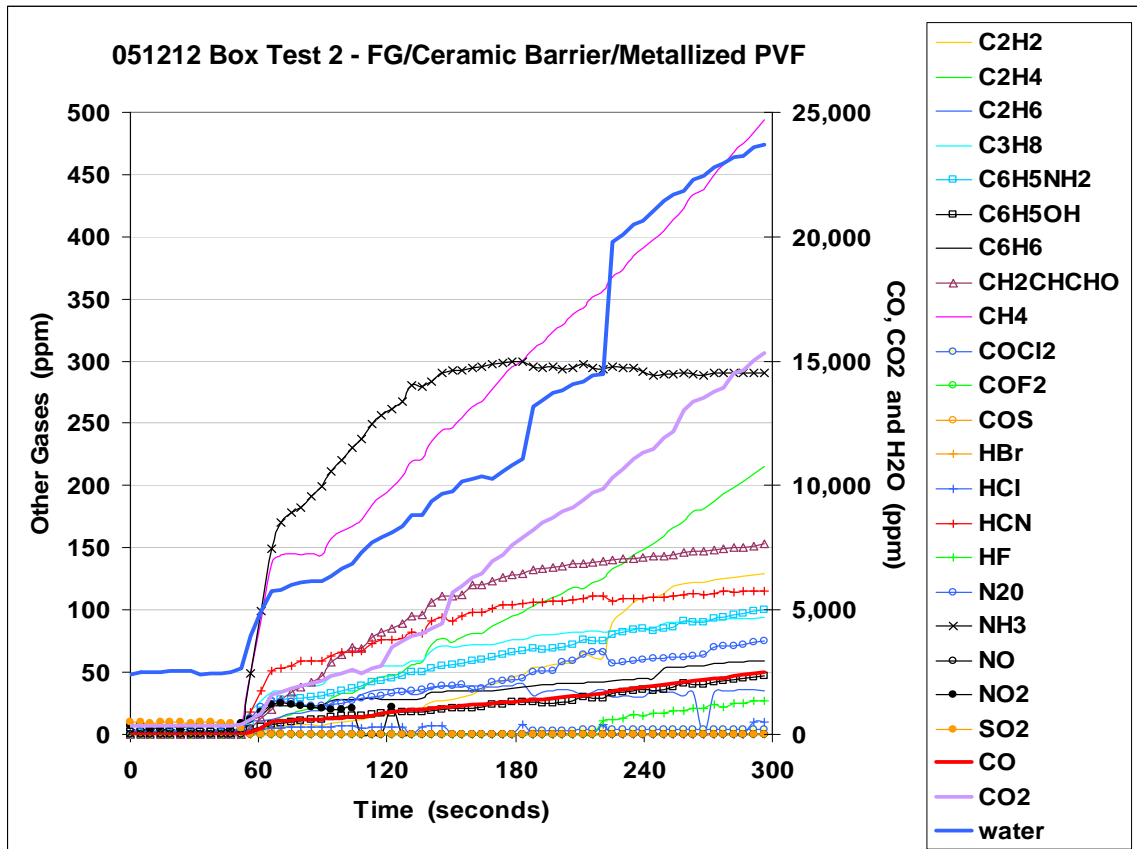
Figure 23. The FTIR Spectra of the Three Material Systems Obtained 5 Minutes Into the Test

FIBERGLASS AND DOT-PRINTED CERAMIC INSULATION SYSTEM. A thin, fire-resistant layer of ceramic fiber material known as Nextel™ was evaluated. Developed by the 3M Company, Nextel ceramic oxide fibers are continuous, polycrystalline metal oxide fibers suitable for producing textiles without the aid of other fiber or metal inserts. The polycrystalline fibers are typically transparent, nonporous, and have a diameter of 10-12 μm. The continuous nature and flexibility of the ceramic oxide fibers allows them to be processed into a variety of textile shapes and forms using conventional weaving and braiding processes and equipment. In this particular arrangement, a nonwoven mat of dot-printed ceramic was tested to determine its effectiveness when used as an additional barrier to the existing fiberglass insulation.

The ceramic barrier and fiberglass insulation batts were encapsulated with the standard metallized PVF moisture barrier film. The ceramic barrier was installed on the outboard face of the insulation batts (within the film) to form a flame propagation barrier between the external flames and the interior of the fuselage. The insulation batts, along with the ceramic barrier, were clamped in place around the perimeter; the clamping also held the ceramic barrier in place. This arrangement was very effective, preventing burnthrough for nearly 5 minutes. During a posttest inspection, it was observed that the majority of the ceramic barrier had remained in place. However, in one area, it was also clear that the barrier had opened and allowed flames to penetrate.

Nextel ceramic fibers are composed of AlO_3 , SiO_2 , and B_2O_3 . The moderate HCN levels may be due to binders in the ceramic paper and or fiberglass insulation.

Figure 24 illustrates the gas concentration histories obtained from the fiberglass/ceramic barrier insulation system test. Five minutes into the test, concentrations of 2,525 ppm CO , 15,308 ppm CO_2 , 116 ppm HCN, 291 ppm NH_3 , 153 ppm CH_2CHCHO , 59 ppm C_6H_6 , and 101 ppm $\text{C}_6\text{H}_5\text{NH}_2$, and 48 ppm $\text{C}_6\text{H}_5\text{OH}$ were measured; and the CO_2/CO ratio was 5/1. At 5 minutes, the H_2O concentration reached 2.4%. The concentrations of hydrocarbons measured in the test box were far below the level to cause a flashover event. The lower explosive limit (LEL) of CH_4 , C_2H_6 , and C_3H_8 are 5.0%, 3.0%, and 2.1%, respectively. The 5-minute concentration of CH_4 was 494 ppm. The 5-minute concentrations of C_2H_2 , C_2H_4 , and C_2H_6 were 130, 215, and 35 ppm, and C_3H_8 was 94 ppm. C_6H_6 , $\text{C}_6\text{H}_5\text{OH}$, and $\text{C}_6\text{H}_5\text{NH}_2$ were 59, 48, and 101 ppm, respectively. A rough estimate of the percentage of LEL of the hydrocarbons as propane can be made by assuming that it is proportional to the number of carbon atoms. Table 1 provides the contribution of C1, C2, C3, and C6 hydrocarbons to the percentage of LEL as propane (as determined by FTIR analysis). Collectively, the combined effect of these gases was only about 5% of the propane LEL. The source of nitrogen for HCN, NH_3 , etc. is possibly a binder, which may be present in the insulation system.



FG = Fiberglass

Figure 24. Concentration Histories of Nextel Insulation System Box Test Obtained by FTIR Analysis

Table 1. Computation of the Percentage of the LEL as Propane at 5 Minutes for a Fiberglass/Ceramic Barrier Insulation System Test

Number of Carbons	Gases	Concentration as Propane (ppm)	Percentage of LEL as Propane
1	CH ₄	494/3 = 165	(165/21,000)*100 = 0.78
2	C ₂ H ₂ , C ₂ H ₄ , C ₂ H ₆	(130 + 215 + 35) *2/3 = 254	(254/21,000)*100 = 1.21
3	C ₃ H ₈ , CH ₂ CHCHO	94 + 153 = 247	(247/21,000)*100 = 1.18
6	C ₆ H ₆ , C ₆ H ₅ OH, C ₆ H ₅ NH ₂	(59.3 + 47.5 + 101)* 6/3 = 207	(207/21,000)*100 = 1.98
1, 2, 3, and 6		165 + 254 + 247 + 415 = 1080	(1080/21,000)*100 = 5.14

THE PAN INSULATION SYSTEM. Another series of tests was conducted using an oxidized PAN fiber supplied by TexTech Industries of North Monmouth, Maine. The material was similar to the heat-stabilized PAN material supplied by the Orcon Corporation (Curlon[®]) that was tested previously in the full-scale rig. Curlon is a heat-treated, oxidized polyacrylonitrile fiber. Curlon contains about 70% carbon, 20% nitrogen, and 10% oxygen. The materials are unique because they could potentially be used as drop-in replacements for the current fiberglass insulation (i.e., they possess some qualities similar to fiberglass for the intended use in aircraft applications, such as noise attenuation). The PAN material supplied by TexTech was extremely effective at resisting flame penetration for at least 5 minutes.

Early large spikes in the concentrations of NH₃, HCN, CH₄, and H₂O can be seen at 106 seconds in figure 25. This is consistent with the high position of the sample probe and the auto-accelerated exothermic stabilization reaction of remaining unstabilized PAN. The PAN was probably not fully stabilized. Above 220°C, these stabilization reactions are spontaneous, with a rapid uncontrolled release of heat [8]. During stabilization, dehydrogenation reactions evolved H₂O, decarbonization reaction evolved CO₂, and nitriles are evolved as HCN. The carbonization reactions of stabilized PAN can be accelerated by this initial large exotherm resulting in the early spikes. It can be seen that the spikes are not primarily due to a pressure event, since the spike was very minor for many of the gases.

In the 300°-350°C region, the main reactions of the carbonization process occur on the chain ends, generating NH₃, H₂O, CO₂, HCN, and low molecular weight nitriles. In the 700°-1000°C range, substantial amounts of HCN, NH₃, N₂, and water with lesser amounts of low molecular weight nitriles, CO₂, CO, H₂ and methane are expected to be generated [8].

Figure 25 illustrates the gas concentration histories obtained by FTIR for the PAN insulation system test. Five minutes into the test, concentrations of 0.46% CO, 1.18% CO₂ (1.6% with the CO₂ analyzer), 467 ppm HCN, 377 ppm NH₃, 266 ppm SO₂, 79 ppm C₆H₆, 70 ppm C₆H₅NH₂, 56 ppm CH₂CHCHO, 39 ppm COS, 0 ppm NO, and 22 ppm HF were measured. Note that this FTIR method is not accurate for CO₂ (See previous FTIR method section.). The CO₂/CO ratio was 2.6, and a concentration of 18.2% oxygen and 1.2% H₂O was measured. The hydrocarbon composition at 5 minutes based on FTIR analysis is shown in table 2. Observed THCs were 0.06% as propane, based on the FTIR measurements, and 0.08% as propane (table 2), based on

the THC analyzer, or 3% to 4% of the propane LEL, respectively. The concentration histories obtained by gas analyzers are illustrated in figure 26.

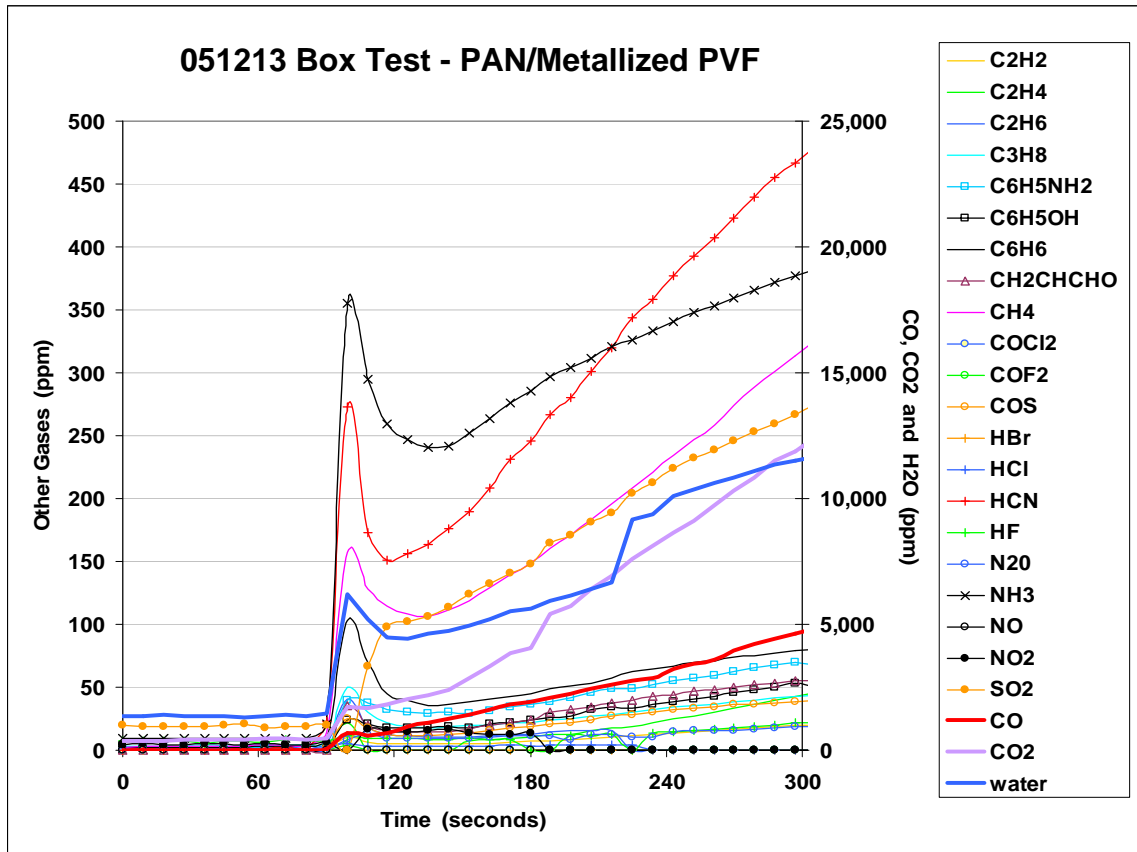


Figure 25. Concentration Histories of PAN Insulation System Box Test Obtained by FTIR Analysis

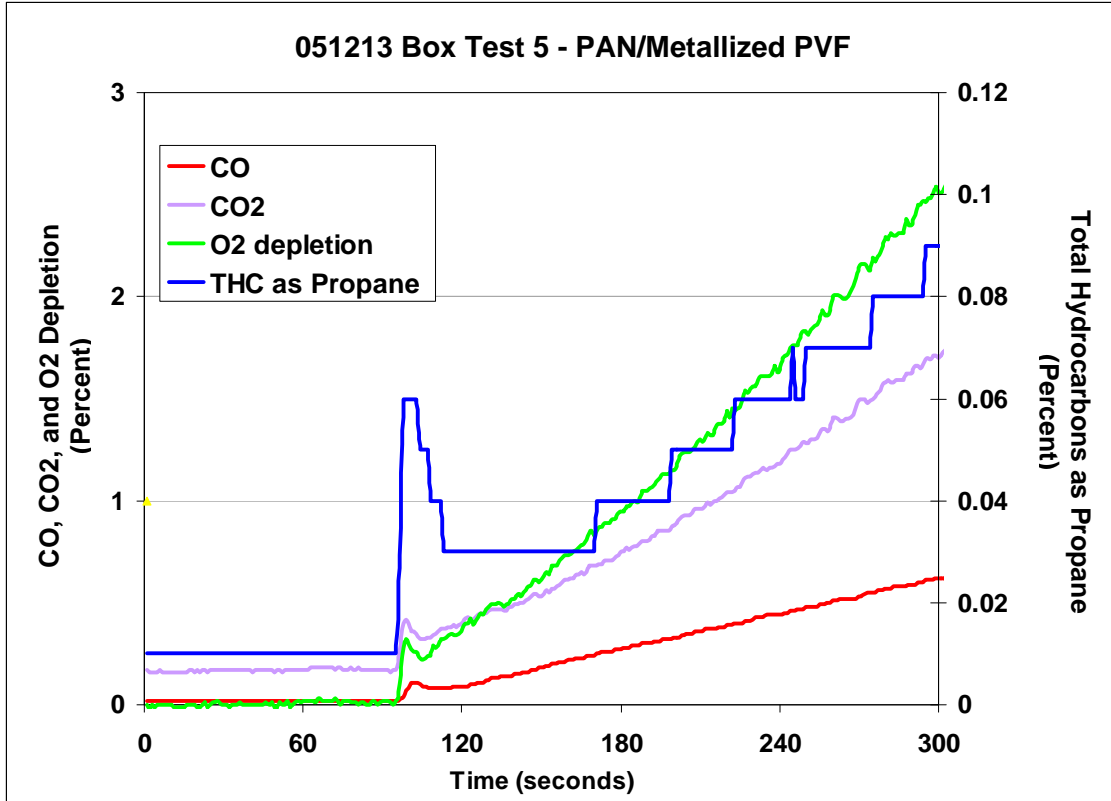


Figure 26. Concentration Histories of PAN Insulation System Box Test Obtained by Gas Analyzers

Table 2. Computation of the Percentage of the LEL as Propane at 5 Minutes for PAN Insulation System Test

Number of Carbons	Gases	Concentration as Propane (ppm)	Percentage of LEL as Propane
1	CH ₄	313.9/3 = 104.6	(104.6/21,000)*100 = 0.050
2	C ₂ H ₂ , C ₂ H ₄ , C ₂ H ₆	(20.2 + 342.7 + 0) * 2/3 = 41.9	(41.9/21,000)*100 = 0.20
3	C ₃ H ₈ , CH ₂ CHCHO	42.9 + 55.5 = 97.4	(97.4/21,000)*100 = 0.46
6	C ₆ H ₆ , C ₆ H ₅ OH, C ₆ H ₅ NH ₂	(78.7 + 52.7 + 70.7) * 6/3 = 403.2	(403.2/21,000)*100 = 1.92
1, 2, 3, and 6	All the above	104.6 + 41.9 + 97.4 + 403.2 = 647.2	(647.2 / 21,000)*100 = 3.08

INITIAL LABORATORY-SCALE EVALUATION OF ACMs (NO ENCLOSURE).

The ACM was purchased from Toray Compositions (America). It had been cured at 350°C for 6 hours. The fibers are embedded in an epoxy matrix. Decomposition products have been characterized by Jones and Pedrick [9]. The predegradation products HCl and HF probably arise from reagent impurities. The postdegradation products are H₂S, CH₄, and HCN. The major identified products in the primary degradation are C₆H₅NH₂, H₂O, C₃H₈, and SO₂. C₆H₆, C₆H₅NH₂CH₃, and C₆H₅NHCH₂CH₃ have also been identified. Because the ACM incorporated an epoxy resin system, it was initially speculated that a combustible mix of gases could be

produced and emitted from the backface side of the test sample. Since the steel box enclosure did not incorporate any type of pressure relief blowout panels to alleviate a spike in pressure in the event of rapid combustion of these gases, the initial burnthrough test was run using the standard open-frame sample holder. The heated gas-sampling line was initially located centrally on the unexposed side of the test rig, near the top of the test sample holder, to capture any gases generated from the back surface of the test sample. Relatively constant concentrations of H₂O, CO₂, SO₂, C₆H₅NH₂, NH₃, HCl, CH₄, and C₃H₈ were observed for the open-frame test (figures 27 and 28). These gases may be, in part, decomposition products of the kerosene burner. Except for H₂O and CO₂, the measured gas concentrations were all below 10 ppm.

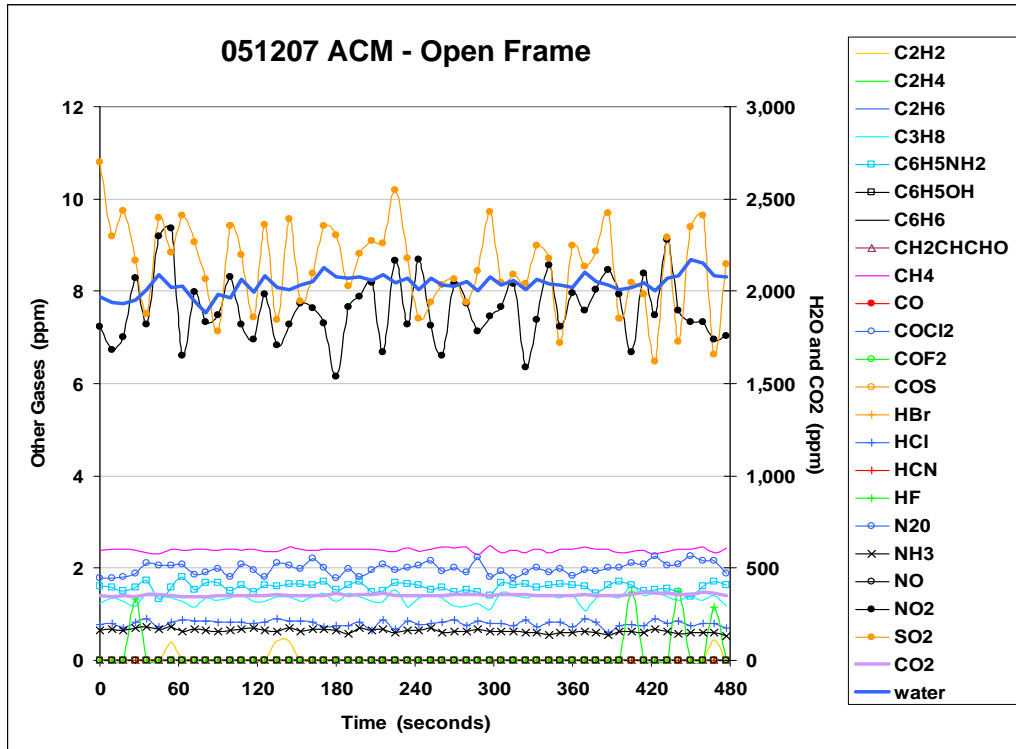


Figure 27. Concentration Histories of the Backface of ACM With no Enclosure Obtained by FTIR Analysis

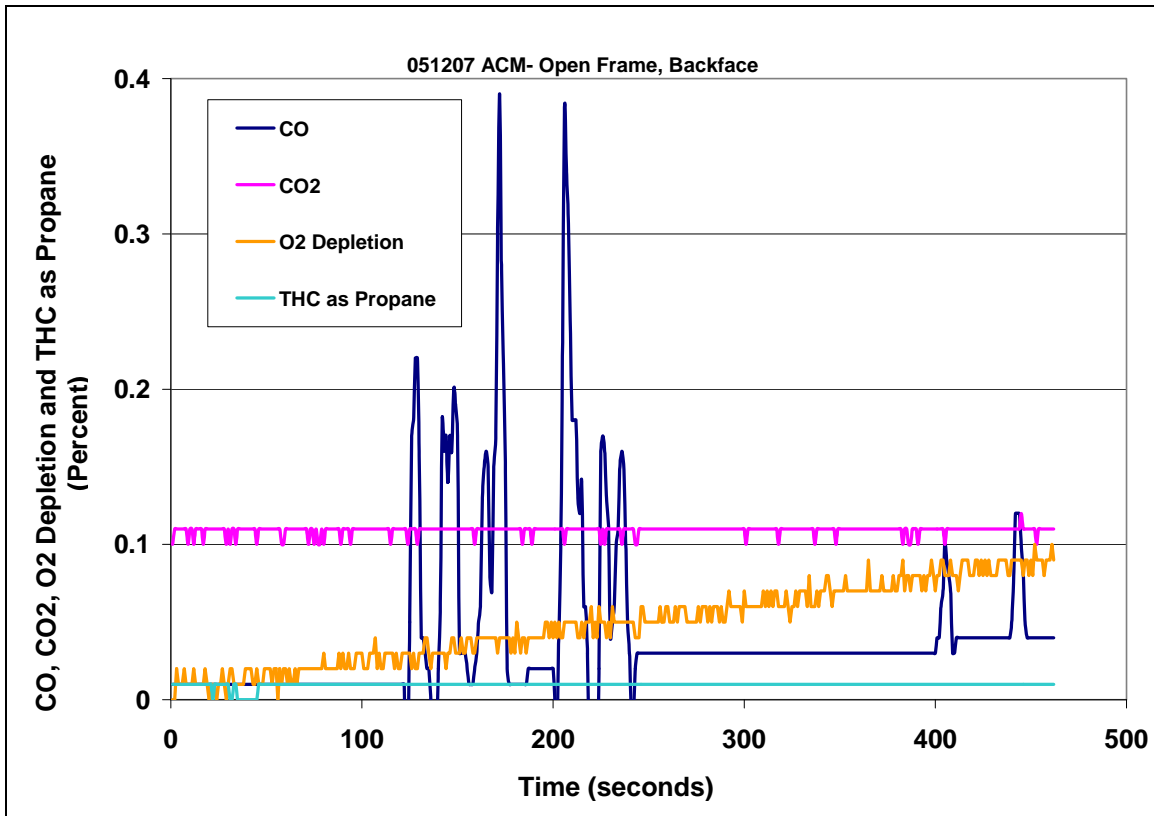


Figure 28. Concentration Histories of the Backface of ACM With no Enclosure Obtained by Gas Analyzers

Once the burnthrough test was underway, it was apparent that very little decomposition gases were being emitted from the backface of the test sample. The ACM test sample was exposed to the burnthrough test burner for over 7 minutes, at which point the burner flame was turned off. A concentrated area on the backface of the test sample began to smoke at approximately 4 minutes into the test. To gauge the composition of the gases within the smoke plume, the test probe was repositioned into the plume at 7 minutes. The gas analysis equipment was run for an additional 90 seconds before termination (figure 29). There was no significant change in gas concentrations.

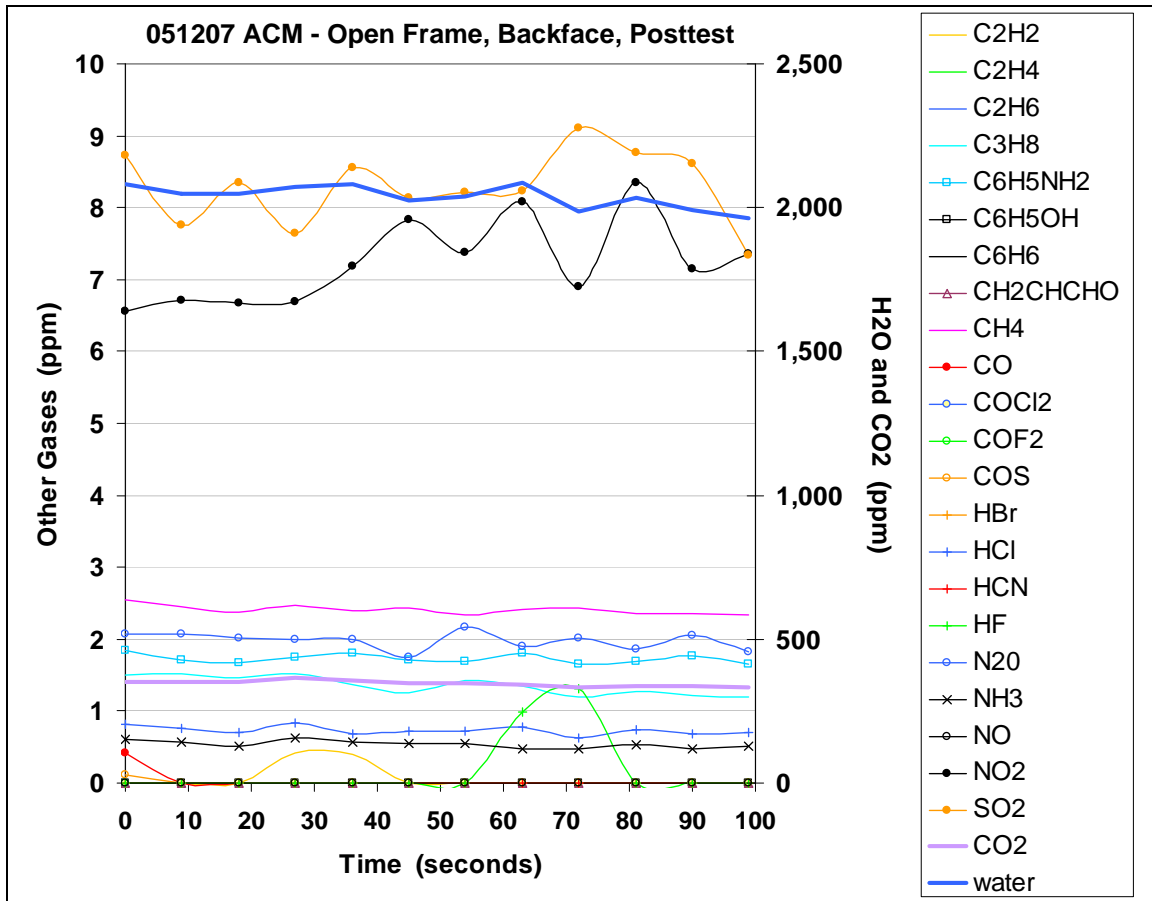


Figure 29. Posttest FTIR Analysis of Backface of ACM in Open Frame With Probe Positioned Closer to Sample (Within Smoke Plume From Backface of Panel)

EVALUATION OF ACMs.

Following the open-frame test of the ACM, in which a very small amount of constituents were released from the nonexposed side of the test sample, it was agreed that a safe test could be run using the steel box enclosure. To ensure safety, a 6- by 6-inch blowout panel was installed in the box to prevent a catastrophic overpressure in the event of gas ignition. The composite panel was mounted into the recessed area of the box opening, and the securing flange and bolts were installed and tightened.

Five minutes into the test, concentrations of 56 ppm CO, 464 ppm CO₂, 0 ppm HCN, 4 ppm NH₃, 34 ppm SO₂, 9 ppm C₆H₆, 0.8 ppm COS, 0 ppm CH₂CHCHO, and 6 ppm C₆H₅NH₂ were measured (figures 30 and 31). The observed CO₂/CO ratio was 8.3. A concentration of 21% O₂ and 3714 ppm H₂O was measured at 5 minutes. The hydrocarbon composition at 5 minutes based on FTIR analysis is shown in table 3. Observed THCs were as high as 0.008% as propane, or 0.4% of the propane LEL).

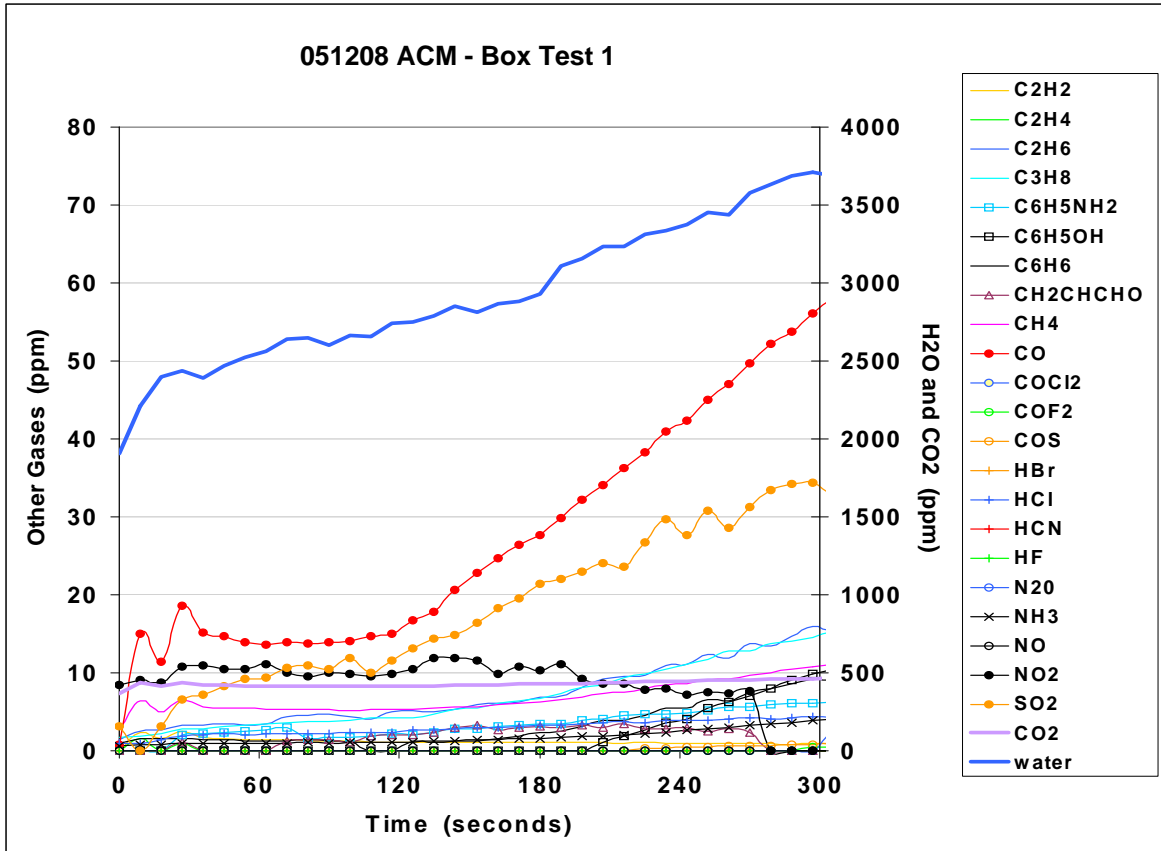


Figure 30. Concentration Histories of ACM Box Test 1 Obtained by FTIR Analysis

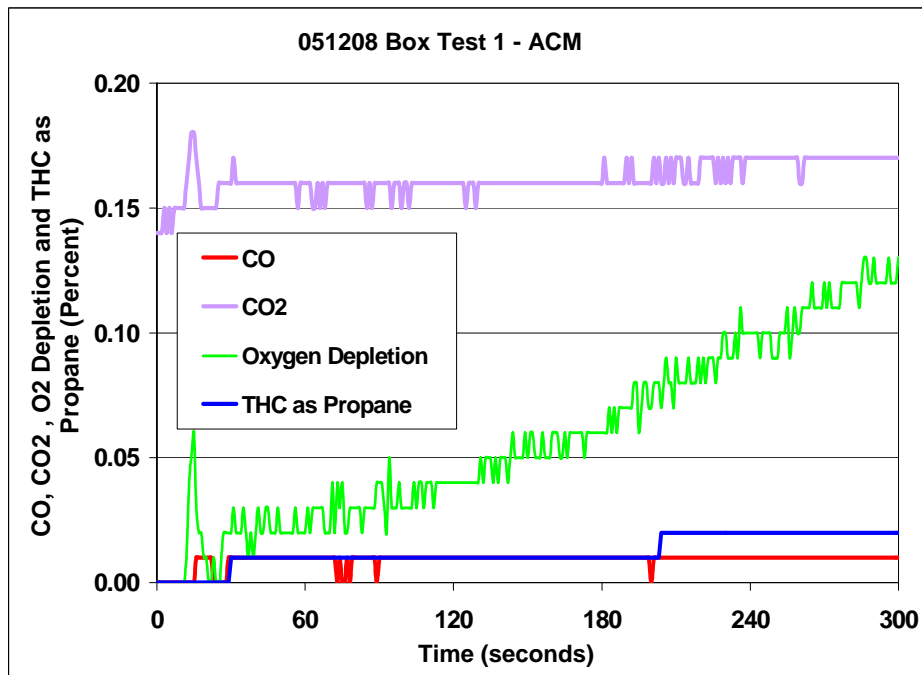


Figure 31. Concentration Histories of ACM Box Test 1 Obtained by Gas Analyzers

Table 3. Computation of the Percentage of the LEL as Propane at 5 Minutes for ACM Test

Number of Carbons	Gases	Concentration as Propane (ppm)	Percentage of LEL as Propane
1	CH ₄	10.7/3 = 3.6	(3.6/21,000)*100 = 0.017
2	C ₂ H ₂ , C ₂ H ₄ , C ₂ H ₆	(0.9 + 0 + 16.0) *2/3 = 11.2	(11.2/21,000)*100 = 0.053
3	C ₃ H ₈ , CH ₂ CHCHO	14.5 + 0 = 14.5	(14.5/21,000)*100 = 0.069
6	C ₆ H ₆ , C ₆ H ₅ NH ₂ , C ₆ H ₅ OH	(9.3 + 9.8 + 6.1)* 6/3 = 50.3	(50.3/21,000)*100 = 0.24
1, 2, 3, and 6		3.6 + 11.2 + 14.5 + 50.3 = 79.6	79.6 (21,000)*100 = 0.379

A second test was run using the ACM, to confirm the previous test result (figure 32), and the results were reasonably consistent.

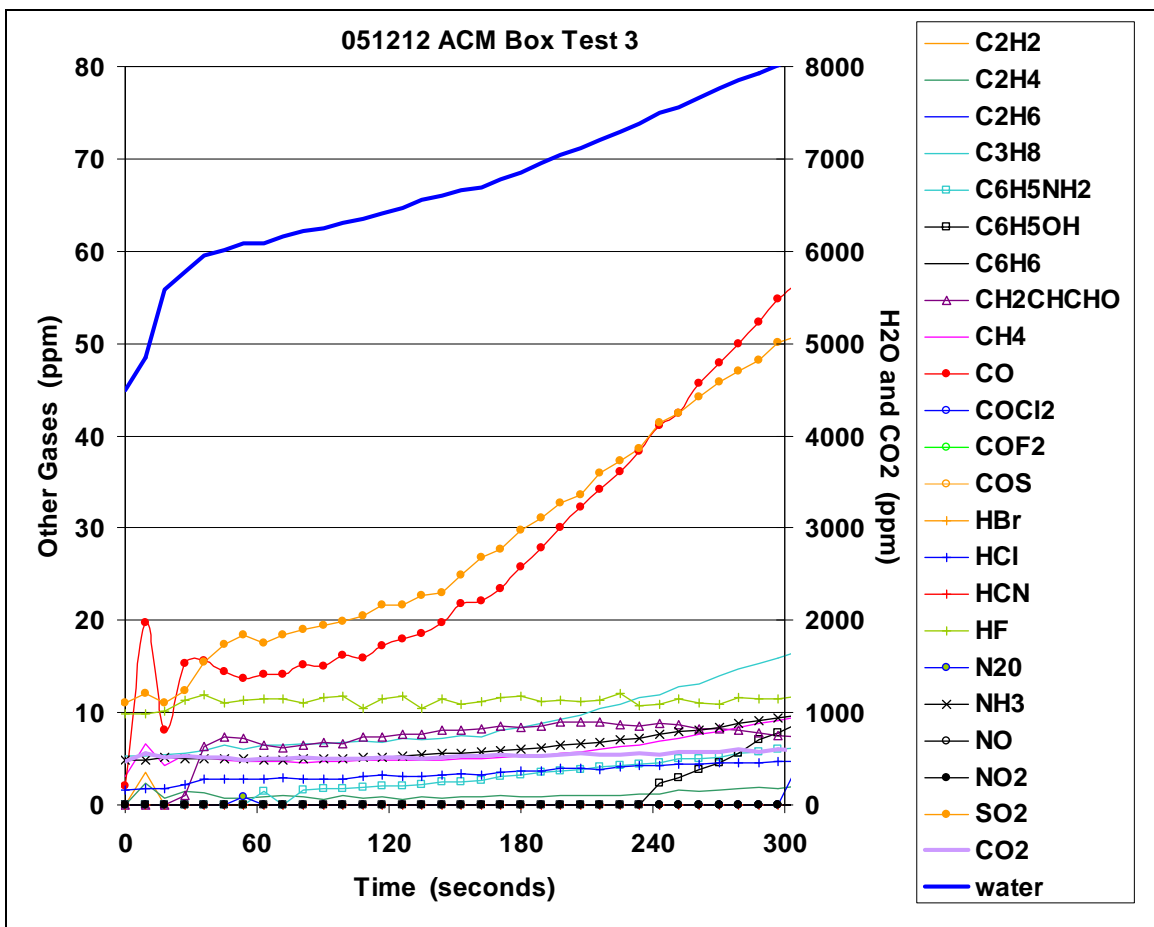
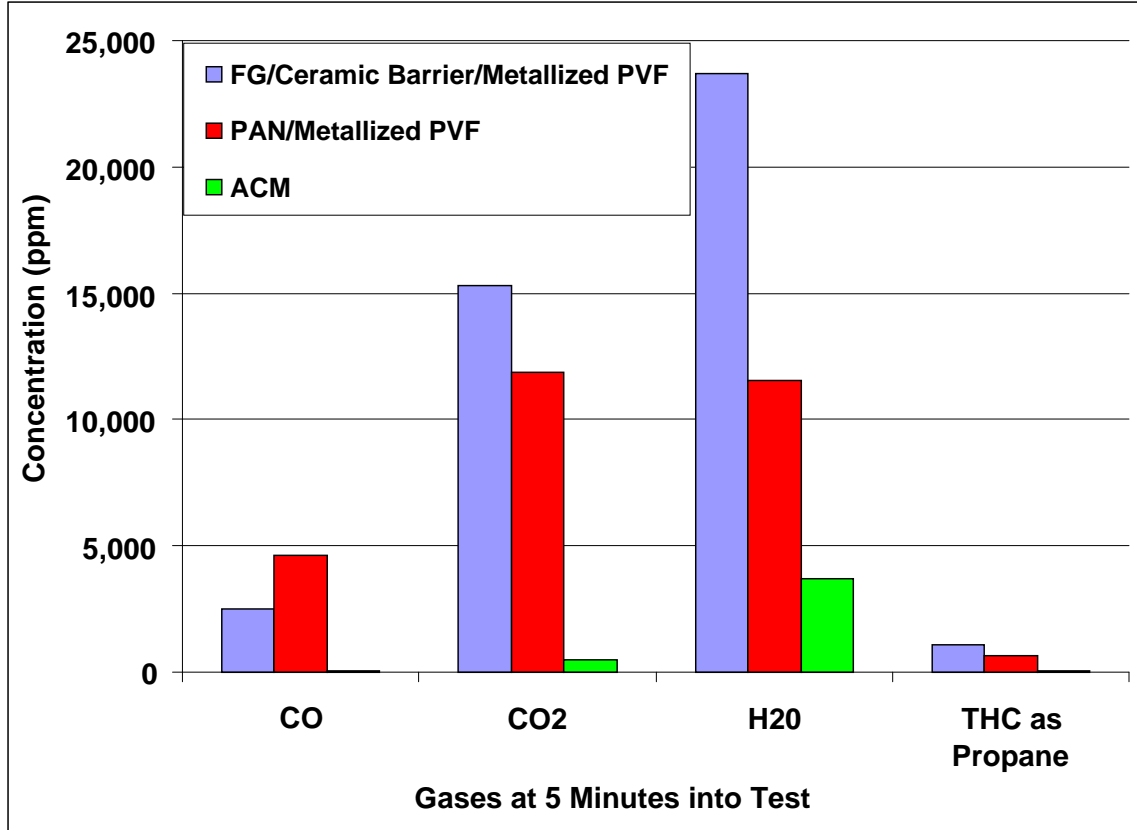


Figure 32. Concentration Histories of ACM Box Test 3 Obtained by FTIR Analysis

COMPARISON OF TEST RESULTS.

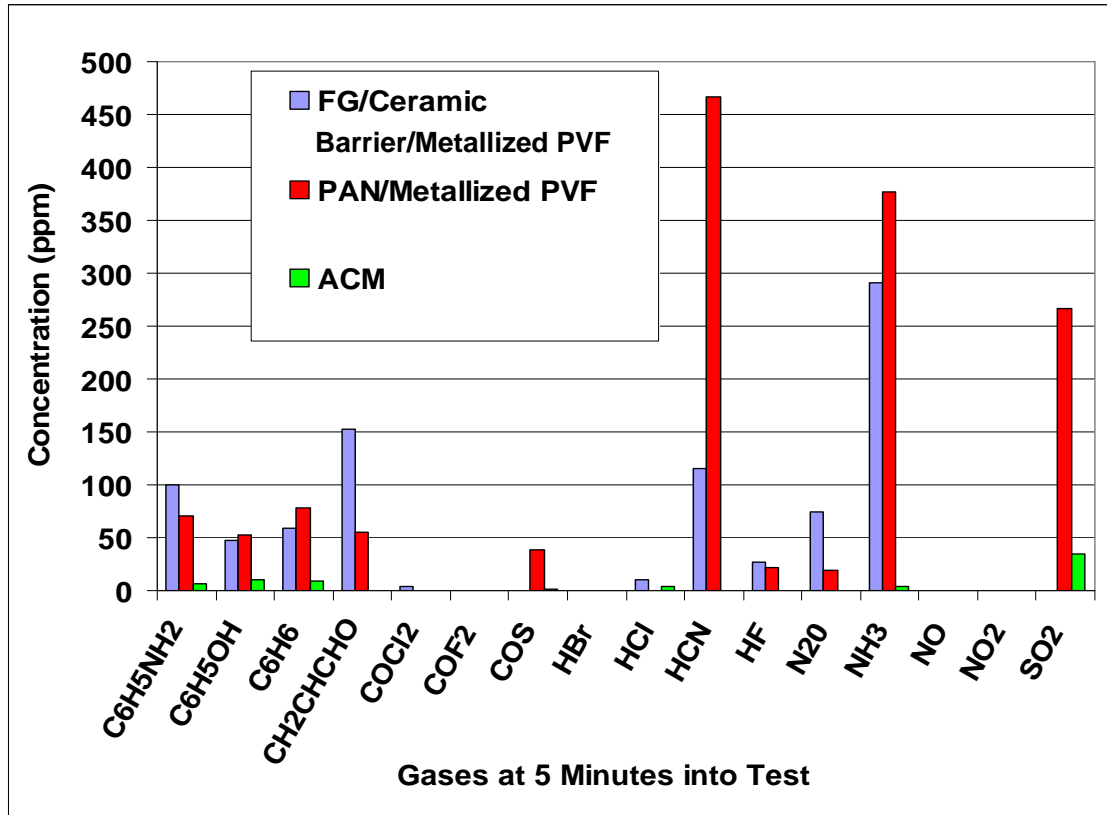
Figures 33 and 34 compare the FTIR-derived gas concentrations at 5 minutes for the three material systems tested in this study. As shown in figure 33, both the ceramic barrier and the PAN insulation systems generated greater quantities of CO, CO₂, H₂O, and THC than the ACM.

Similarly, as shown in figure 34, all other gases measured also resulted in higher quantities during tests of the ceramic barrier and PAN insulation systems, as compared to the ACM.



FG = Fiberglass

Figure 33. Comparison of Box Test Concentrations Obtained at 5 Minutes by FTIR for the Three Material Systems: CO, CO₂, H₂O, and THC



FG = Fiberglass

Figure 34. Comparison of Box Test Concentrations Obtained at 5 Minutes by FTIR for the Three Material Systems: Other Gases

SUMMARY

A laboratory-scale test method was developed for evaluating the products of combustion emitted from fuselage/insulation samples designed to remain intact during exposure to a simulated external fuel fire. The equipment used in the test (an oil-fired burner and a 4- by 4- by 4-foot steel cube box used to mount representative test samples) effectively simulated the desired postcrash fire condition. The cube box also served as an enclosure to collect and analyze emitted gases during fire exposure.

Test samples representing several fuselage constructions were tested to measure the emission of combustion gases from the nonexposed side when subjected to a simulated jet fuel fire. Two fuselage configurations were initially evaluated, consisting of aluminum skin and accompanying insulation materials that met the new FAA burnthrough standard: a ceramic-based, lightweight barrier in conjunction with standard fiberglass batting, and a heat-stabilized PAN fiber. These configurations were primarily run to establish a baseline of the amount and type of gases emitted during exposure for comparing other fuselage/insulation samples. In addition, a third fuselage construction was tested consisting of a prototype structural composite material (without thermal acoustic insulation). It was determined that the prototype, multi-ply, structural composite material produced minimal quantities of smoke, toxic gases, and hydrocarbons during a 5-minute

exposure. Approximately 7 plies of the 13-ply test panel were penetrated by the fire. Overall, the aluminum skin/insulation configurations generated higher gas concentrations than the composite material during the 5-minute exposure.

CONCLUSIONS

These tests demonstrated that the equipment provided a suitable test method for evaluating potential hazardous gases emitted from a variety of fuselage/insulation samples. The method of clamping the test samples in place onto the box enclosure proved to be effective at eliminating the collection of combustion by-products produced by the burner flame itself. This was important, as the intent was to sample only gases that would emanate from the unexposed side of the test samples during exposure, simulating a continuous fuselage under realistic conditions. The intrusion of burner by-products into the box enclosure would adversely impact the gas analysis. In addition, a higher confidence level in the data for carbon monoxide and carbon dioxide was gained by using two independent and distinctly different types of analyzers (Fourier Transform Infrared analyzer and nondispersive infrared analyzer).

The test method could be used to evaluate the potential toxicity of insulation constructions and innovations that meet the new burnthrough test requirements, to ensure that harmful gases will not result inside the fuselage, despite the high burnthrough performance associated with a particular system. It also could be used to evaluate the toxic contribution of the basic fuselage structure whenever a nonmetallic material is used as the primary component.

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