



APPENDIX AVAILABLE ON THE HEI WEB SITE

Communication 17

Advanced Collaborative Emissions Study (ACES) Phase 3A: Characterization of U.S. 2007-Compliant Diesel Engine and Exposure System Operation

Joe L. Mauderly and Jacob D. McDonald

Appendix B. Protocol

Note: Appendices Available on the Web may appear in a different order than in the original Investigators' Report, and some remnants of their original names may be apparent. HEI has not changed the content of these documents, only the letter identifier.

Appendix B was originally Appendix A

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APPENDIX A

Protocol

PROTOCOL

PHASE 3A: CHARACTERIZATION OF ENGINE-EXPOSURE SYSTEM OPERATION, AND POTENTIAL ANIMAL EXPOSURE ATMOSPHERES

Advanced Collaborative Emissions Study (ACES)

LRRI Protocol Number: FY08-147

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1.0 OBJECTIVES

The work described in this protocol comprises the portion of the ACES program at LRRI that bridges between the commissioning of the engine facility and the conduct of the animal exposure study. The objectives of this portion of the program are to:

- a) Compare the composition of diluted emissions in an exposure chamber at LRRI with comparable data from the same engine and an identical exposure chamber at Southwest Research Institute (SwRI);
- b) Fine tune the dilution-exposure system and confirm stable operation;
- c) Generate temperature and compositional data sufficient for selecting the dilution conditions and resulting animal exposure concentrations for Phase 3B;
- d) Produce a report of results, to serve as a basis for decision-making; and
- e) Assess system performance to enable development of criteria for validation of the entire exposure system in a subsequent protocol.

2.0 BACKGROUND

ACES Phase 3 includes a development of the engine facility (under a separate contract), conducting generation trials and producing data leading to selection of animal exposure conditions (Phase 3A), and conducting the animal study (Phase 3B). This protocol describes the approach to accomplishing Phase 3A. The activities encompassed by Phase 3A are those described as “Tier 1” and “Tier 2” in the original (July 31, 2006) proposal. Phase 3A begins by constructing and commissioning the engine facility laboratory by Affiliated Construction Services (ACS). Upon successful commissioning of the laboratory, information that provides preliminary data for finalization of the inhalation protocol and compares atmosphere measurements to data generated in Phase 1 (emissions characterization) will be developed. This protocol will end with approval of a report that serves as an adequate basis for selecting the final exposure conditions for Phase 3B. After final exposure conditions are defined, a second protocol will be developed to assess and validate the entire exposure system according to pre-defined performance criteria.

This protocol describes key elements of the test cell commissioning and the plan for characterization of test atmospheres under a range of dilution and operating conditions. The key steps of the protocol are 1) commissioning and shake-down of the engine lab facility, 2) development and testing of engine performance with three steady-state modes, the federal test procedure cycle, and the ACES engine cycle, 3) evaluation of emissions characteristics from the cycles, and 4) development of test atmosphere characterizations relevant to the final inhalation protocol. KEY STUDY PERSONNEL

Principal Investigator: Dr. Joe L. Mauderly is the Principal Investigator of the ACES program at LRRI, and has overall responsibility for the elements of the conduct of the program that are under the control of LRRI. He maintains close liaison with the HEI Project Manager, and is the primary point of contact for the program. Dr. Mauderly’s position as Vice President of LRRI ensures top management attention to the program.

Study Director: Dr. Jacob D. McDonald will be the study director for Phase 3A, having primary responsibility for the conduct of the research and generation of results. This

responsibility includes the general strategy for accomplishing the work, establishing specific procedures, supervising technical personnel, ensuring that necessary standard operating procedures (SOPs) and study-specific procedures (SSPs) are developed and approved, ensuring that data are processed through appropriate quality control and quality assurance (QC/QA) reviews, and developing the final report of the Phase.

Database Manager: Dr. Nancy L. Crowley will be responsible for establishing an appropriate database structure and security strategy for data acquired during this Phase, maintaining the database, and providing data files to investigators and the sponsor as requested.

Systems Integration Engineer: Mr. Lonny J. Peet (ACS) will be responsible for oversight of the installation and commissioning of the facility and test equipment. This will include engine and facility operations, data collection, data review and analysis, and training.

Exposure Operations Supervisor: Mr. Richard K. White will be responsible for the day-to-day training and line supervision of the technical staff operating the engine and exposure system.

Analysis of Organic Compounds: Dr. Barbara Zielinska (Desert Research Institute) will provide sampling media and conduct analysis for speciated gaseous and semi-volatile organics.

Analysis of Inorganic Compounds: Dr. Judith Chow (Desert Research Institute) will provide sampling media and conduct analysis for carbon, inorganic ions, and metals.

3.0 CONFIDENTIALITY

LRRI staff will not disclose raw data or summary results from work performed under this protocol in response to inquiries from third parties, except as directed by the HEI Project Manager. The HEI Project Manager will be informed of all such requests. LRRI will not disclose results of work under this protocol in any written or oral external communication without advance permission from the HEI Project Manager.

LRRI will maintain the confidentiality of the identity of the test engine. LRRI will not disclose the identity of the test engine, except by permission from the HEI Project Manager. LRRI will not allow visitors view the engine or obtain any information that might reveal the identity of the engine. An exception will be personnel representing the engine manufacturer and its designated internal and local technical support organizations. The engine manufacturer will be responsible for ensuring that their representatives maintain confidentiality. Another exception will be non-LRRI individuals that might be required to repair or maintain non-engine components of the engine or exposure facility. LRRI will be responsible for ensuring that such individuals maintain confidentiality. In written and oral communications concerning the project (such as the HEI Annual Conference), LRRI will use the engine description preferred by HEI.

4.0 QUALITY ASSURANCE

For facility and engine system commissioning a systematic series of tests will be iteratively conducted to achieve target performance criteria. Several measurements will be conducted, including assessments of air flow, liquid flow, pressure, and temperature. As appropriate, these measurement devices are calibrated and/or verified against primary standards by the manufacturer. Once the engine commissioning is completed, a Standard Operating Procedure will be developed for operation of the engine and associated systems. All characterization

procedures will be conducted according to LRRI study specific Standard Operating Procedures (SOPs). These SOPs define procedures for conduct of the work, including calibrations, tolerance, precision/accuracy verification, sample handling, field and laboratory blank requirements, sample storage, sample shipping, analysis, and data handling. The SOPs also will define requirements for quality control and data verification required prior to reporting.

5.0 ENGINE AND EXPOSURE FACILITY

Figures 1 and 2 show schematics of the engine laboratory and dilution tunnel. The laboratory is equipped with a Dyne-systems Dymond-Series 550 horsepower rated Alternating Current (AC) dynamometer. This is equipped with an Inter-Loc V Modular Multi-Loop Controller. The engine will be operated with fuel and lubricant provided by the sponsor. The exhaust transits through a 2007 compliant after-treatment system provided by the engine manufacturer. The dilution tunnel is not a constant volume system. Rather, the tunnel operates at a constant pressure that is maintained by the presence of a supply air by-pass that allows excess air to be dumped to the engine room. The maximum combustion air flow is approximately 1300 cubic feet per minute (CFM). The nominal dilution tunnel flow is 3000 CFM. Tunnel dilution air and combustion air are Charcoal and HEPA filtered.

Figure 3 shows a schematic of the aerosol extraction point, secondary/tertiary dilutions, and the inhalation exposure chamber. Aerosol is extracted from the dilution tunnel through an air-vac pump. Aerosol then transits through a flow-through muffler prior to a series of two by-pass and dilution addition steps. The distance from the extraction point to the exposure chamber is approximately 6 meters. The transit line is composed of 5 cm diameter stainless steel tubing. The secondary and tertiary dilution air is medical grade compressed air.

The extraction points for aerosol characterization from the chamber are shown on the front panels of the exposure chamber in Figure 3. The majority of the chamber sampling will be conducted from the middle port. Samples for particle characterization will be conducted with the analyzers or collection media as close as practical to the extraction point. Gas analyzers will reside on a platform above the exposure chambers.

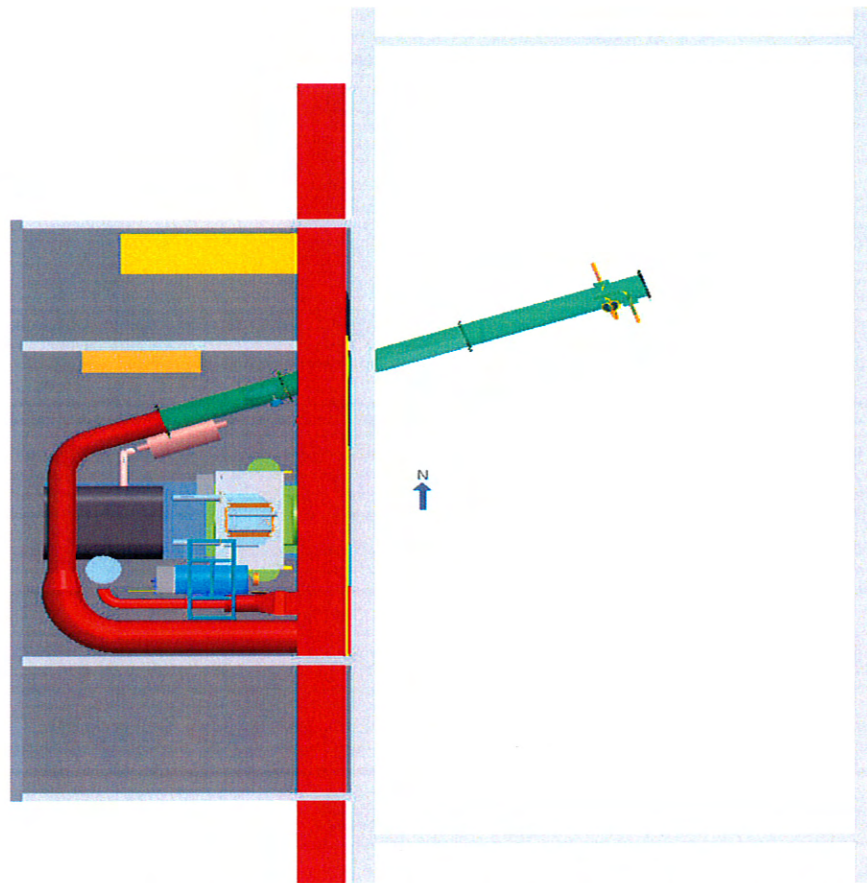


Figure 1. Overlay of engine laboratory and dilution tunnel. The tunnel portions shown in red are upstream from engine exhaust mixing. The exhaust mixing portion is green and the extraction point for the exposure chamber distribution system is at the far North-East corner. . The charge-air-cooler is shown in grey. The engine is shown in black, and the fuel delivery system in blue. The dynamometer is shown in light-green.

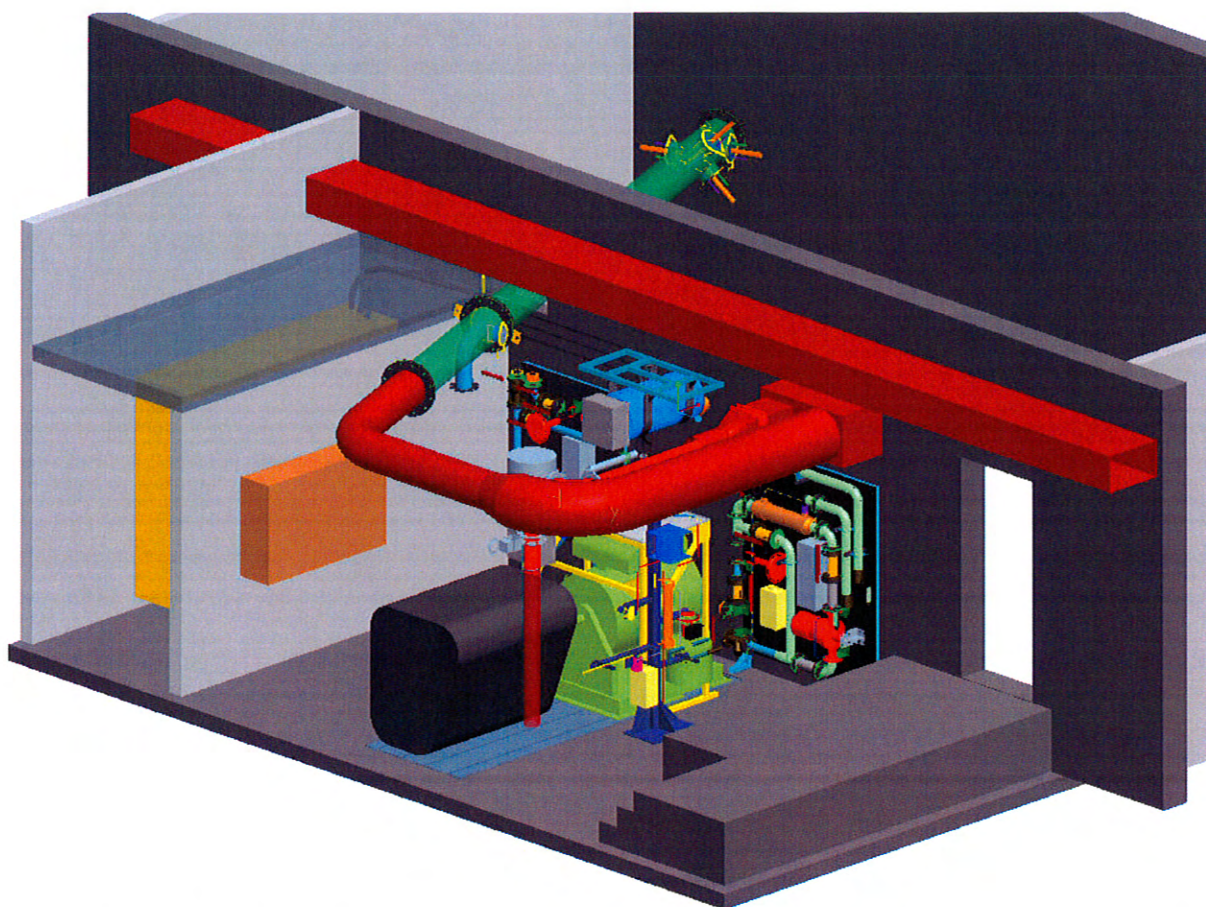
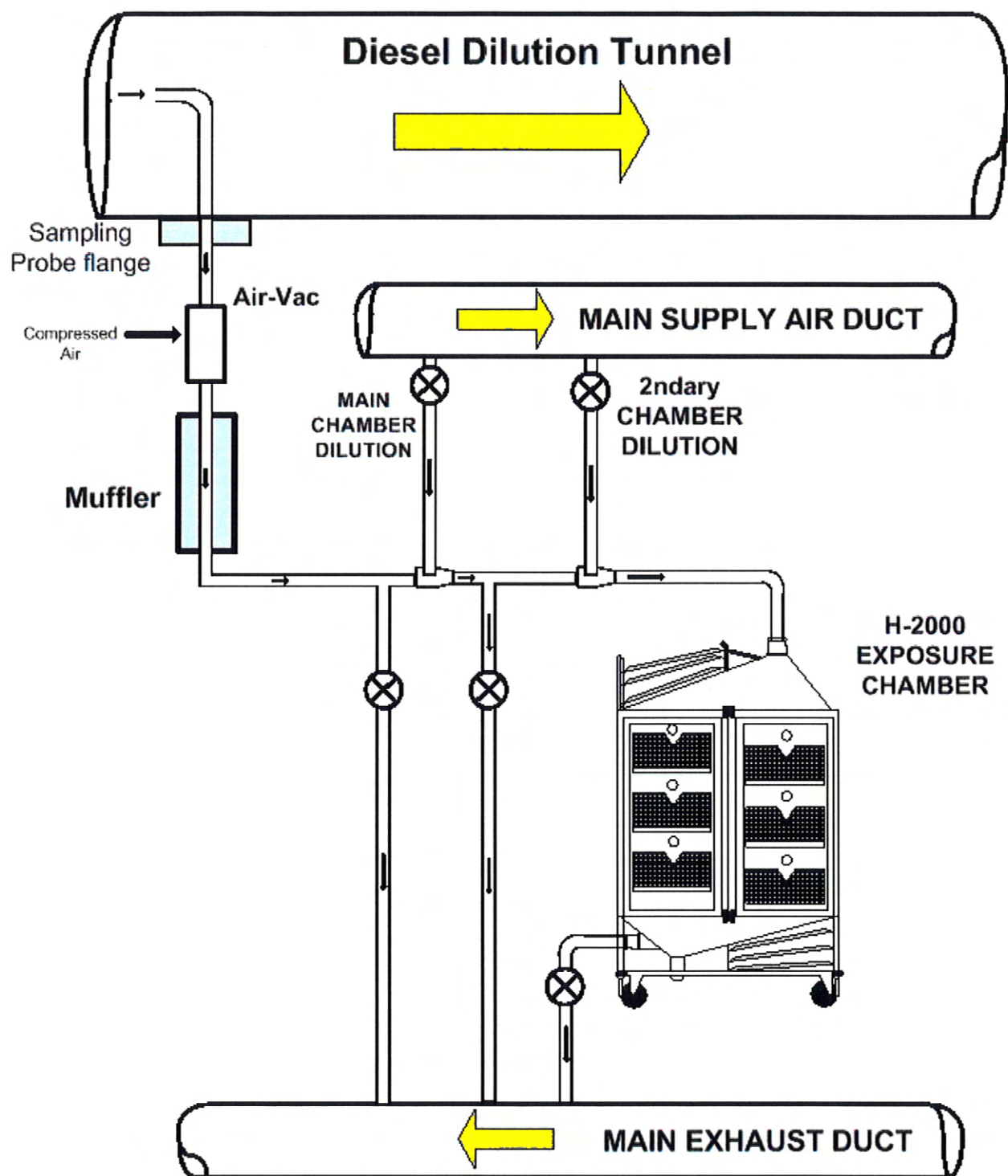


Figure 2. Overlay of engine laboratory and dilution tunnel. The tunnel portions shown in red are prior to engine exhaust mixing. Green shows the exhaust mixing section and the take-off point for the exposure chamber distribution system. The engine is shown in black, and the fuel delivery system in blue. The dynamometer is shown in light-green.



(Note: Drawing is not to scale)

Figure 3. Schematic (not to scale) of secondary and tertiary dilution system prior to inhalation exposure chamber. Diluted exhaust is extracted through the sampling probe using an air-vac pump. Aerosol will transit through a flow-through muffler prior to a series of two by-pass and dilution legs.

6.0 COMMISSIONING OF ENGINE FACILITY

The objectives of the initial commissioning phase are to ensure 1) proper operation of the mechanical systems, 2) proper operations of the electrical systems, 3) proper operation of the engine on the federal test procedure cycle, and 4) verification of cycle performance for the ACES engine cycle. Facility testing will be conducted with a surrogate engine of the same make and model as the ACES Phase 3 engine. This engine will be operated under the steady state and federal test procedure cycles for an initial system shake-down. Subsequently, the ACES phase 3 engine will be installed and the engine cycles (including the ACES cycles) will be verified prior to the emissions characterizations described below.

Table 1 below summarizes the specific tests that will be conducted as part of the engine facility commissioning. The table identifies specific systems to be tested, the objective and general scope of the test, and (where appropriate) the design criteria that will be evaluated. We note that each system has a number of components that will be evaluated. These individual components integrate to the final design criteria requirements that are defined below. We do not call out each of the individual performance checks as part of the design criteria.

The initial shake-down may require iterative testing of the control systems to adjust towards the specified design criteria. Performance to the design criteria will be evaluated to ensure that the actual operating conditions are within specification. Specifications for temperatures, pressures and flows will be within 10% of the design criteria. Engine power data (speed, torque, horsepower) will be maintained within 5% of the design criteria. For evaluation of the engine cycle an analysis of engine power, torque, speed, air flow rates, fuel flow rates, temperatures and pressures will be compared to target values by a regression analysis. Cycle precision will be determined by a minimum of three repeat cycles that enable an assessment of the coefficient of variation for these parameters. The target coefficient of variation for target versus actual cycle performance is 5%. This will be conducted for Modes 1, 3 and 5 of the ISO 8178 steady state cycle, the FTP cycle, and the ACES Phase 3 cycle.

Table 1. Summary of facility systems and test equipment that will be tested for the associated Design Criteria.

System	Objective	Design Criteria
Jacket Water – engine block cooling	Verify proper installation and function of heat exchangers, pumps, control valves, current switches, temperature and pressure indicators & elements.	Engine Outlet: 200 °F Engine Inlet: 180 °F Chilled Water Flow Rate: 97 gallons per minute (GPM) Outlet: 70 °F Inlet: 55 °F
Charge Air Cooling – turbo compressed air temperature control	Verify proper installation and function of heat exchangers, pumps, control valves, current switches, temperature and pressure indicators & elements, balancing valves, and adjustment for proper flow rates of process fluids including tuning of system control loops	Engine Flow Rate Max: 1330 cubic feet/minute (CFM) Engine Outlet: 420 °F Engine Inlet: 80 °F Chilled Water Flow Rate: 102 GPM Outlet: 82 °F Inlet: 77 °F
Combustion Air – engine intake air	Verify proper installation and function of heat exchangers, control valves, temperature and pressure indicators & elements, flow measurement devices, and adjustment for proper flow rates of process fluids including tuning of system control loops	Engine Flow Rate Max: 1330 CFM Re-heat Coil Outlet/Engine Inlet: 77 °F Re-heat Coil Inlet: 59 °F Chilled Water Flow Rate: 3.18 GPM

Table 1. Summary of facility systems and test equipment that will be tested for the associated Design Criteria. (Concluded)

System	Objective	Design Criteria
Dilution Air – mixing tunnel air to be mixed with the exhaust	Verify proper installation and function temperature and pressure indicators & elements, flow measurement devices, and adjustment for proper flow rates of process fluids	Dilution Air Flow Rate Max: 3000 CFM Inlet Temperature: 59 °F Sample Zone Temperature Max: 375 °F
Cell Ventilation – test cell air conditioning	Verify proper function of existing fans, dampers, temperature switches, and adjustment for proper flow rates of process fluids	Max Temperature: 120 °F Flow Rate: 8500 CFM
Fuel Conditioning – supply, recirculation, temperature, pressure, and measurement	Verify proper installation and function of heat exchangers, pumps, control valves, safety valves, safety interlocks, current switches, level controllers, level switches, level transmitters, temperature and pressure indicators & elements, balancing valves, and adjustment for proper flow rates of process fluids including tuning of system control loops	Engine Fuel Flow Rate Range: 26–120 gal/hr Engine Fuel Inlet Pressure: -6 to 3 psig Engine Fuel Inlet Temperature: 104 °F
Engine Load and Speed Control – AC dynamometer, speed and torque measurement & control	Verify proper installation and function of ac motor and variable frequency drive, cooling fans, temperature and pressure indicators & elements and include the calibration of torque, speed, and throttle measurement and control devices using vendor provided means and methods including tuning of system control loops	Engine Power Max: 500 hp Engine Peak Torque: 1800 ft-lb Engine Max Speed: 2300 RPM
Data Acquisition and Control System	Verify proper installation and function of input and output modules, relays, power supplies, computer hardware, and software Verify and calibrate all analog channels including pressure & temperature inputs using vendor provided means and methods Verify proper function of safety systems and PLC logic including tuning of system control loops Verify proper function of the requested test cycles	Requested capabilities: Recording of facility and performance data typical of a transient test cell ACES 16 hr test cycle FTP test cycle Modes 1, 3, & 5 of ISO 8178 8-mode test cycle
Engine Cycle Performance	Verify performance of engine cycles by characterizing target versus actual engine power, torque, speed, air flow rates, fuel flow rates, temperatures and pressures.	Within 5% coefficient of variation comparing target versus actual. Within 5% precision for repeat of same engine cycles.

7.0 CHARACTERIZATION OF EXHAUST AND INHALATION EXPOSURE SYSTEM

Table 2 summarizes the tests, objectives and number of trials for each set of tests for the characterization of the ACES engine. The characterization will be conducted subsequent to the change-out of the surrogate engine for the Phase 3 engine. Once in place, exhaust characterization of the regulated pollutants will be measured for modes 1, 3, and 5 of the steady state cycle, the FTP cycle, and the ACES phase 3 cycle. Assessment of detailed chemical composition, including all of the measurements summarized in Table 3 will be conducted at the minimum dilution meeting animal welfare requirements. The rationale and a description for each test is provided below. All of the testing will utilize the 16 hour transient cycle that was developed for the ACES program. Measurements will all be conducted in a single H-2000 exposure chamber, using one that is currently installed for use at the highest exposure concentration. This was the type of chamber used at SwRI. Chamber environmental conditions (temperature and humidity) will be monitored during each test.

Table 2. Summary of tests, objectives and number of trials for each set of tests for the characterization of the ACES engine.

Test	Objective	No. of Trials	Notes
1. Exhaust characterization on steady state and FTP cycles	Evaluate exhaust characteristics to verify proper engine performance	3/cycle condition	Only regulated gases, particle mass and size distribution
2. Determine composition and environmental conditions at 40 x dilution rate	Compare SWRI exposure chamber composition to LRRI exposure chamber composition	3	SWRI chamber characterization used the 40 x dilution rate. Only regulated gases, particle mass and size distribution
3. Determine minimum allowable dilution rate	Define highest concentration in exposure chamber at which temperature within the chamber is < 27 °C (target 24 ° average temperature)	Iterative	Dilution rate must result in proper temperature. NO ₂ and CO may also be considered as limiting factors.
4. Determine chamber atmosphere composition at minimum allowable dilution rate	Verify repeatability of composition and environmental profile at proposed minimum allowable dilution	3	Will measure all components in Table 3.
5. Determine chamber performance at minimum allowable dilution rate	Pre-validation to characterize T90, chamber homogeneity and repeatability	3	Only regulated gases, particle mass and size distribution
6. Determine chamber atmosphere composition at 10 x minimum dilution	Define atmosphere proportionality with dilution rate for key constituents	3	Only regulated gases, particle mass and size distribution
7. Determine particle number count and size distribution in chamber and prior to exposure chamber	Assess particle changes as a result of inhalation exposure chamber and aerosol transit lines	3	Determine with steady state and FTP cycles

7.1 Exhaust Characterization on Steady State and FTP Cycles

Exhaust characterization will be conducted to measure NO_x, NO₂, CO, non-methane hydrocarbons, particle number and size distribution after primary dilution from the engine. This will be conducted in modes 1, 3 and 5 of the steady state engine cycle and with the FTP cycle. Regeneration of the exhaust after-treatment system will be initiated manually and completed before initiation of steady-state and FTP cycles used for exhaust characterization. Exhaust will be extracted from the primary dilution tunnel and further diluted with clean compressed air to achieve a temperature at the filter face of < 90 °F. This secondary dilution will occur as close to the primary dilution tunnel as practical. Samples will be collected for a duration sufficient to achieve enough sample to make the measurement. This will be defined by the filter sample requirements. FTP cycle measurements will be conducted to ensure that the start and end of the sample collection occurs at the same point in the engine cycle.

7.2 Determine Composition and Environmental Conditions at 40 x Dilution

The dilution rate in the exposure chamber will be set to 40 x to match the rate used to assess chamber atmosphere composition at SWRI. The dilution rate will be defined by the ratio of engine-out carbon monoxide (CO) to exposure chamber CO. CO will be collected simultaneously at both locations over a 4 hour segment of the 16 hour transient cycle. The dilution rates will be determined 3 times for each condition.

Chamber composition and environmental conditions will be assessed in detail by all measurements defined in Table 3. Speciated hydrocarbons (gaseous and semi-volatile) will be collected and analyzed in two of the three tests. These measurements will be conducted during the entire 16 hour cycle.

7.3 Determine Minimum Allowable Dilution Rate

The maximum achievable exposure atmosphere concentration will be defined by the dilution required to achieve animal welfare standards for temperature ($75 \pm 3^{\circ}\text{C}$) and humidity (< 70%) averaged throughout the 16 hour transient cycle. The ACES Oversight Committee may also consider NO₂ and CO as determinants of the minimum required dilution.

The initial target dilution rate will be 10 x. If this dilution does not meet environmental requirements, the rate will be adjusted iteratively to define the minimum dilution that enables environmental conditions within the required limits. Each test will be conducted over an entire 16 hour cycle. If 10 x does not meet the requirements, the next increment will be 20 x. Dilution will subsequently be adjusted in ± 5 increments (e.g., 25 x or 15 x) until a suitable dilution is identified.

7.4 Determine Composition of the Chamber Atmosphere at the Minimum Allowable Dilution Rate

Inhalation exposure atmosphere characterization will be conducted at the lowest allowable dilution ratio (highest test atmosphere concentration). Chamber composition and environmental conditions will be assessed in detail by all measurements defined in Table 3. Speciated hydrocarbons (gas and semi-volatile) will be collected and analyzed in two of the three tests. Measurements will be conducted during the entire 16 hour transient cycle.

7.5 Determine Chamber Performance at Minimum Allowable Dilution Rate

7.5.1 Exposure Chamber Aerosol Distribution

Uniformity of aerosol concentration in the exposure chambers will be measured. The uniformity test will verify the homogeneity of the test atmospheres for both gaseous (based on NO and NO₂) and particulate (Dekati mass monitor) fractions. A schematic diagram of the H-2000 exposure chamber and sample ports is presented in Figure 4. Four sampling ports (two positions, one at the front and one at the back for each of four ports located on the doors of the H-2000 chamber) will be used. Samples will be taken by a defined protocol to allow system homogeneity to be defined. The total variation of aerosol concentration is defined as the coefficient of variation of samples taken in series at each of the sampling ports during steady-state operation, and the temporal variation is the coefficient of variation between the data referenced to one sampling port that is sampled continuously throughout the test. The spatial variation can be calculated:

$$CV(\%)_{\text{spatial}}^2 = CV(\%)_{\text{total}}^2 - CV(\%)_{\text{temporal}}^2,$$

under the assumption that the spatial and temporal variations are independent of each other.

7.5.2 Determination of T90

It is anticipated that the dilutions within the exposure chambers will be monitored and defined daily based on the concentrations of NO. Importantly, the concentration of nearly all components of the test atmosphere will be changing throughout the engine cycle. Therefore, the value of a T₉₀ (time to 90% of a target concentration) that is typically used to define exposure system characteristics is not plausible to define precisely during the 16 hr engine cycle. In order to define a value for time to equilibrium, the T-90 will be measured with the engine operated in steady-state. The target will be defined based on the time to system equilibrium (steady-state concentration) within the exposure chamber.

7.5.3 Residual Aerosol Concentration During Non-exposure Hours

During nonexposure hours the exposure systems will be flushed with clean air; they will not be drawing air from the dilution tunnel. In order to define any background or re-entrainment of gaseous and particulate components during non-exposure hours, we will measure regulated pollutants in the exposure chambers for the entire 8 hour nonexposure period on days for which the system has been operated for multiple preceding days.

7.5.4 Exposure System Stability

Exposure system stability will be characterized during the triplicate operations at the proposed minimum dilution.

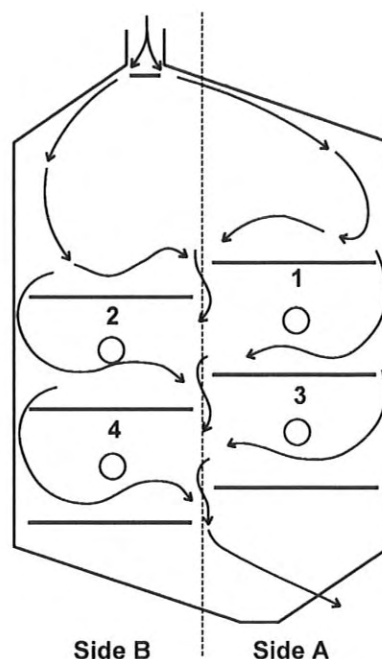


Figure 4. H-2000 Exposure chamber schematic showing sampling ports and flow dynamics.

7.6 Determine Composition in the Chamber at 10 x Minimum Dilution

Particle mass, size and the regulated gases will be assessed at 10 x the minimum dilution rate. These tests will allow assessment of the proportionality of atmosphere composition with increases in dilution. Concentrations of measured components will be compared at the 100 x dilution and the proposed minimum dilution.

7.7 Determine Particle Number Count and Size Distribution in Chamber and Prior to Exposure Chamber

Particle number and size distribution will be measured from the dilution tunnel after a secondary dilution to decrease temperature. This will be compared against particle number and size distribution made in the inhalation exposure chamber under the same engine operating conditions. The tests will be conducted for a minimum of five minutes at each of the steady state operating modes, and a minimum of two FTP cycles for the transient cycle testing.

8.0 ATMOSPHERE CHARACTERIZATION

The measurements and analytical techniques to be utilized during Tier 1 testing are summarized in Table 3. The procedures for performing these measurements are described following the table. Note that all real-time measurements will be recorded with 1 second resolution.

Table 3. Summary of measurements, measurement conditions, and analytical techniques used to characterize exposure chamber atmospheres for Tier 1 testing.

Analysis	Collection Device	Collection Media	Analytical Instrument
Gravimetric Mass (PM _{2.5})	Cyclone/Aluminum in-line filter holder	Teflon membrane filter	MB
Continuous Mass	Dekati Mass Monitor	NA	NA
Nitric Oxides (NO _x , NO ₂)	Chemiluminescence Analyzer	NA	NA
Carbon Monoxide/Carbon Dioxide	Infrared Analyzer	NA	NA
Non-methane hydrocarbons	Heated Flame Ionization Detector	NA	NA
Sulfur Dioxide	Ultraviolet pulsed fluorescence	NA	NA
Organic/Elemental Carbon	Aluminum in-line filter holder	Heat-treated quartz filter	TOR
Speciated Metals	Teflon filter holder	Teflon membrane filter	XRF/ICPMS
Speciated Organic Compounds			
Volatile hydrocarbons (C ₁ -C ₁₂)	Volatile organic sampler	Electropolished canister	GCMS
Volatile carbonyls	Volatile organic sampler	DNPH cartridge	LC/UV
Semi-volatile/fine particle organics (1)	Tisch environmental PUF sampler	TIGF filter/XAD-4	GCMS

Table 3. Summary of measurements, measurement conditions, and analytical techniques used to characterize exposure chamber atmospheres for Tier 1 testing. (Concluded)

Analysis	Collection Device	Collection Media	Analytical Instrument
Size Distribution/Mass			
0–1 micron microns mass diameter	Dekati Mass Monitor	NA	NA
0.5–20 microns aerodynamic distribution	Aerodynamic Particle Sizer	NA	NA
~5–500 nm particle number distribution/mass	Fast Mobility Particle Sizer	NA	NA

NA: not applicable; TIGF: teflon impregnated glass fiber; XAD-4: sorbent resin; MB: microbalance; TOR: thermal/optical reflectance; IC: ion chromatography; XRF: X-ray fluorescence; ICPMS: inductively coupled plasma mass spectrometry; LCUV: liquid chromatography/ultraviolet detection; GCMS: gas chromatography/mass spectrometry.

8.1 Sample Collection

Sample collection for tunnel emissions measurements will be obtained as close as practical to the tunnel after a secondary dilution step that will bring temperature to acceptable limits. Exhaust measurements will then be obtained from a small plenum (< 1 second residence time) that will allow simultaneous collections of particle mass on a Teflon membrane filter or extraction to gas and particle analyzers. For the exposure chamber characterization, the Protocol is aimed at determining the composition of the atmosphere at the rodents breathing zone. The exposure chambers contain multiple sample ports that allow samples to be taken directly from the breathing zone of the animals (immediately above the wire cages at different levels in the chamber). The multi-port design is used to characterize the homogeneity of the exposure atmosphere throughout the chambers. With the exception of the Teflon membrane filter for metals and the filter/XAD-4 cartridge for semi-volatile organics (see below), all chamber samples will be collected from the breathing zone through sample ports shown in Figure 2. Samples will be pulled through ~ 12" long ¼" diameter stainless steel probes. Chamber uniformity assessments will confirm that samples can be collected in parallel ports and be representative of the same atmosphere.

We have observed in previous studies that a sample flow above 10 L per minute from the in-chamber ports can disturb the homogeneity of material inside the chamber by altering flow kinetics. For some of the analyses (especially for low concentration analytes such as metals and semi-volatile organics), a larger sample flow is required to obtain the mass of material necessary for trace analysis. In this case, larger volume samples may be collected from the chamber exhaust in a stainless steel plenum. Prior to sample collection at the down-stream location, particle number and mass measurements will be taken simultaneously in the chamber and at the downstream sampling plenum to ensure there is not substantial bias due to sample loss.

All real-time samples will be initiated a minimum of 10 minutes prior to the start of a test, which is initiated by turning on the engine/dynamometer. This will enable data to be collected to establish baseline conditions in the chamber prior to exhaust. Integrated (filters, cartridges, canisters) samples will be turned on immediately after the engine cycle is initiated. All samples will be monitored or collected throughout the 16 hour transient cycles.

8.2 Particulate Matter Mass

8.2.1 Gravimetric Analysis

Gravimetric (differential weight) analysis will be conducted using 47-mm Teflon filter (TEFLO, Pall-Gelman). Filter weights will be measured gravimetrically using a Mettler MT5 microbalance. A static discharger will be used prior to weighing the filters to avoid any interference from electrical charge on the filters. Filters will be pre- and post-conditioned (equilibrated) for 24 hours at 23 °C (± 5) and 40% (± 10) relative humidity prior to weight.

8.2.2 Real-Time Mass and Size Distribution

Real-time PM (and PM size distribution) will be measured using a Dekati mass analyzer (DMM-230) and fast mobility particle sizer. Real-time measurements will be initiated a minimum of 10 minutes prior to the initiation of a test to collect data on background PM in the exposure chambers.

Particle size will be measured by a combination of a DMM-230 (0–1.2 microns), a fast-response differential mobility analyzer (approximately 5–500 nm) and an aerodynamic particle sizer (0.5–20 microns). All analyzers will be operated under default settings, assuming unit density spherical particles. The Dekati analyzer will provide near real-time data on mass median aerodynamic diameter. The fast-response differential mobility analyzer (DMA, Model 3091 Fast Response Particle Sizer [FMPS] Spectrometer, TSI, Inc., St. Paul, MN) also will be used to give particle number size distribution (in real-time) with enhanced resolution for particle size (resolution 1 second).

8.3 Chemical Characteristics of PM

8.3.1 Elemental and Organic Carbon Mass

Elemental and organic carbon mass will be determined at Desert Research Institute on pre-baked quartz fiber filters by the modified Interagency Monitoring of Protected Visual Environments (IMPROVE) thermal-optical reflectance (TOR) method (Chow et al., 1993).

8.3.2 Organic Carbon Class and Species

Particulate and semi-volatile organic compounds (SVOC) will be collected using zeflour filters followed by XAD-4 resins cartridges. The target analytes will be compounds that were statistically above detection limits during the Phase 1 component of the ACES program. Organic analysis for SVOC will be conducted at Desert Research Institute by gas chromatography/mass spectrometry.

8.3.3 Total Metals and Associated Elements

Metals will be collected on clean Teflon membrane filters and analyzed at the Desert Research Institute by energy dispersive X-ray fluorescence. After EDXRF analysis, the Teflon-membrane filters are returned to their Petri slides and stored under refrigeration until the XRF data validation is completed and indicates that the runs were acceptable. Select samples may also be analyzed by Inductively Coupled Plasma Mass Spectrometry based on results from Phase 1 analysis.

8.3.4 Inorganic Ions: Ammonium (and Ammonia), Sulfate, Nitrate

One-half of the quartz filters (and blanks) that are collected for the carbon analysis will be extracted and analyzed at Desert Research Institute for water soluble chloride, nitrite, nitrate, sulfate and formic and acetic acid by ion chromatography. This extract also will be analyzed for NH_4^+ by the indolphenol colorimetric method. The colorimetric method also will be used to measure NH_3 collected and extracted from a citric acid impregnated filter (NH_4^+). The denuder/coated filter will be used to measure total NH_3 and to estimate the positive artifact of NH_3 that is measured as NH_4^+ on the quartz filter.

8.4 Gases and Vapors

8.4.1 Oxides of Nitrogen, Total Hydrocarbons, Sulfur Dioxide, and Carbon Monoxide

NO_x will be measured using chemiluminescent analysis (Teledyne Model 200 series; Ecophysics 700 series). CO and CO_2 concentrations will be determined using a nondispersive infrared gas analyzer (California Analytical Model 600 series). Total VOCs will be measured using a real-time flame ionization detector (Model 300H, California Analytical Instruments) calibrated against a certified propane standard. SO_2 will be measured by ultraviolet pulsed fluorescence (Thermo Electron Model 43i). Analyzers will be zeroed daily using ultra zero air and calibrated with National Institute of Standards and Technology (NIST)-traceable span gases.

8.4.2 Gas Phase Hydrocarbon Speciation

VOCs (except acids and carbonyls, which are too polar for collection and analysis from canister) will be collected using a custom-designed canister sampler (L. Sheetz Enterprises, Reno, NV). Samples will be collected downstream of a NO_x denuder in a pre-cleaned Summa canister and analyzed within 30 days of collection to ensure accurate characterization of polar compounds that may “stick” to the walls of the canister. The NO_x denuder will reduce NO_x and ameliorate NO_x -VOC reactions that lead to false low concentrations of reactive compounds such as 1,3-butadiene and styrene. Analysis will be conducted at Desert Research Institute by GC/MS.

8.4.3 Analysis of Carbonyl Compounds

Carbonyl compounds will be collected on DNPH-impregnated silica gel cartridges preceded by a commercially available oxidant scrubber and a Teflon membrane pre-filter to remove PM. In order to assess the trapping efficiency of the DNPH cartridge, two cartridges will be used in series, and the back-up cartridge will be analyzed to ensure that all carbonyls are trapped on the first cartridge. Analysis will then be conducted at Desert Research Institute by LC/UV (photo-diode-array).

9.0 DATA ANALYSIS AND REPORTING

Data from data acquisition and control system will be reported in a table with the units described in Table 4. For engine cycle performance the performance will be presented as the coefficient of variation. The report will summarize the target design criteria compared to the results obtained during the commissioning. A pass/fail will be indicated to assess the acceptability of the data relative to the original design criteria.

Table 4. Summary of measured and calculated facility and engine performance data to be reported.

Measured and Calculated Value	Engineering Units
Power	kW
Cycle Power	kW/hr
Speed	RPM
Avg Speed	RPM
Torque	N-m
Cycle Torque	N-m
Throttle command	% open
Fuel mass flow rate	kg/hr
Cycle fuel mass consumed	kg
Intake air flow rate	kg/hr
Cycle intake air mass consumed	kg
Dilution air flow rate	kg/hr
Cycle dilution air mass consumed	kg
Various engine and facility pressures	mm-Hg
Various engine and facility temperatures	°C

Data from the emissions characterization will be provided in units of grams/brake-horsepower hour or the particle count/size distribution statistics will be reported by displaying contoured histograms that portray size as a function of time. Data from the chamber atmosphere will be reported in units of degrees centigrade (temperature), per cent (relative humidity), ppm (gases), ppb (gases), $\mu\text{g}/\text{m}^3$ (particle mass and carbon), or particles/ cm^3 (particle number). Particle size distribution will be reported as median diameter with an associated geometric standard deviation. Individual results from each test will be provided in tabular format. Particle number size distribution plots and real-time data will be plotted. A summary of all of the Tier 1 emissions exposure chamber atmosphere results will be submitted in the form of a report to be reviewed by the ACES Oversight Committee. Based on the results, a go-no go decision for proceeding to inhalation exposures will be made, and final dilution targets will be defined. Subsequently, a system validation protocol will be developed to enable validation of the entire exposure system under final conditions for the Phase 3 studies.