# A Controlled Inhalation Diesel Exhaust Exposure Facility with Dynamic Feedback Control of PM Concentration

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A facility has been assembled that provides a controlled inhalation exposure to freshly diluted and mixed diesel exhaust using a diesel engine under load and a two-stage exhaust dilution system with dynamic feedback control. The concentrations of particulate matter less than 2.5  $\mu$ m in diameter (PM<sub>2.5</sub>), particulate carbon, and gaseous pollutants including carbon monoxide and oxides of nitrogen (NO<sub>x</sub>) have been characterized and the exposure conditions have been found to be both stable and reproducible. Control of the PM<sub>2.5</sub> concentration at intended levels relies on the relatively linear relationship between particle light scattering and exhaust particle mass concentration. While the exposure system does not entirely replicate diesel exhaust conditions in the atmosphere due to the relatively low ratio of nitrogen dioxide to total NO<sub>x</sub>, the fine particulate matter size distributions are quite similar to those of aged diesel exhaust. The facility enables study of the relationship between diesel exhaust and cardiovascular and respiratory health effects in human and animal models.

We briefly describe a facility that provides a controlled inhalation exposure to freshly diluted and mixed diesel exhaust (DE) in order to study the relationship between DE and cardiovascular and respiratory health effects in human and animal models. Unique aspects of this facility, especially for human exposure, are engine operation under load and two-stage dilution system with dynamic control of the exposure concentration. In contrast, other research groups have produced controlled exposures for human inhalation studies using diesel exhaust from an idling truck engine mixed with clean air in a dilution tunnel (Rudell et al., 1994; Salvi et al., 1999; Matsumoto et al., 2006). McDonald et al. (2004a, 2004b) have also reported on the characterization of a diesel exhaust exposure facility for animal inhalation studies using a single-cylinder diesel engine, and a 5.9-L diesel engine coupled to a dynamometer and operated based on the Federal Test Procedure cycle.

# DESCRIPTION OF EXPOSURE ATMOSPHERE GENERATION SYSTEM

The engine/generator combination assembled by Sommers, Ltd. (Tavistock, Ont.) consists of a 2002 model year turbocharged direct-injection 5.9-L Cummins, Inc. (Columbus, IN), B-series diesel engine (model 6BT5.9G6) and a 100-kW generator. The diesel fuel combusted in the engine is standard highway-grade number 2 diesel fuel obtained from local fuel distributors. This fuel meets specifications common to number 2 diesel fuel refined in western Washington State and sold in the region for highway motor vehicle use. Average measured sulfur content of the fuel was 454 ppm through March 2006, at which time the anticipated transition to ultra-low-sulfur diesel

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(ULSD) (U.S. EPA, 2001) was observed in our regular fuel testing results. Analysis of the fuel tank contents in November 2006 indicated a sulfur content of 17 ppm, aromatics content of 25% by volume, and cetane index of 48. Valvoline or Chevron grade SAE 15W-40 motor oil formulated specifically for diesel engines is used for lubricating oil and is replaced every 250 h of operation. The generator-engine set is placed under load with the use of a Simplex (Springfield, IL) model LBS-B-100 load bank. The load bank provides resistive load in stepwise selectable fashion from 0% (0 kW load) to 100% (100 kW load). The load on the engine is normally set at 75% of generator output (75 kW). In addition to the user-specified load from the load bank, the generator also powers the dilution system air compressor, duct heater, and humidifier. These devices typically cycle on and off during normal operation of the dilution system.

The diesel exhaust dilution system consists of a two-step dilution process (see Figure 1). The dilution system is used to mimic the dispersion of diesel exhaust in the atmosphere as described by Kittelson et al. (2002). Exhaust from the engine is sampled from the center of the diesel engine's exhaust pipe downstream of a straight pass-through muffler and is conveyed under positive pressure into the dilution system through the use of an eductor (model TD260LSS, Air-Vac Engineering Company, Inc., Seymour, CT). Primary dilution occurs in two rapid sequential steps: (1) mixing of diesel exhaust in the eductor with filtered compressed air at approximately 1:1 dilution ratio; and (2) immediate downstream mixing of filtered, conditioned air under positive pressure at approximately 14:1 through the use of an Air Blender (model ABG4, Blender Products, Inc., Denver, CO) device. This initially quenched primary mixture then flows through a 3-inch diameter pipe, or "dilution tunnel," for approximately 2 s, where it is then diluted at approximately 27:1 with secondary makeup air. All makeup dilution air is first heated and humidified (thermostat and humidistat conditions:

21°C and 50% relative humidity [RH]) and then passed through both a carbon-impregnated (Air Handler model 14524244, Dayton Electric Mfr. Co., Niles, IL) and HEPA filter (99.99% efficiency). A large Air Blender (model S17C2S) promotes rapid and thorough mixing of the merging air streams.

The well-mixed diluted exhaust stream enters the intake plenum, a 20.4-m<sup>3</sup> box containing vertical aluminum baffles that produce nearly uniform flow before it enters the 116-m<sup>3</sup> exposure chamber through a perforated aluminum wall. The diluted diesel exhaust exits the chamber through a second set of perforated aluminum sheets, moves upward through the exhaust plenum, and is conveyed via an axial fan to an exhaust stack.

The dilution system includes a flow-splitting duct near the end of the dilution tunnel where a portion of the primary dilution mixture can be diverted away from the secondary mixing process. Coarse control over the final dilution mixture is achieved by adjusting a slide gate located in this diversion duct downstream of the flow split, allowing more or less primary dilution mixture to enter the secondary dilution duct. Fine control over the amount of dilution is achieved using a variable-speed in-line electric fan in the diversion duct (see Figure 1).

Due to random, temporal, and occasionally cyclical variations in the diesel generator, the exposure chamber will, unattended, drift out of the required concentration range over the course of a several-hour exposure session. The variability is due to factors both biased (engine warmup and trends in local atmospheric conditions) and cyclical (load bank switching). Constant manual adjustment of the diversion fan can achieve  $\pm 20\%$  control; however, a 10% maximum variation requires an automated system.

To meet these control requirements, a data acquisition and control system (DACS) was designed and implemented using National Instruments (NI) hardware (analog and serial I/O breakout boards connected to analog–digital–analog [A/D/A]



FIG. 1. Schematic of diesel exhaust dilution system with feedback control.

interface cards) and LabVIEW software. The system performs data acquisition for particle and gas analyzers, and stores information in text data files for later relational database input. A Dwyer FC 1000 electronic fan speed controller determines the diversion fan speed using inputs supplied by the NI analog output. Three levels of safety are designed into the system: (1) An over-range alarm provides an audible alert at an operator-set level, (2) the inherent chamber response lag allows sufficient time to remove subjects before levels rise appreciably, and (3) a manual switch provides a controller bypass to allow manual fan control in case of any malfunction.

The 6-min residence time between dilution and the chamber's analyzer inlets and the 10-min moving average output of the tapered-element oscillating microbalance (TEOM)-our continuous benchmark PM2.5 measurement-pose a control challenge. Therefore, rapidly responding particle light scattering measurements (Radiance Research model M903 nephelometer, Seattle, WA) are made at the entrance to the intake plenum to control the diversion fan speed through the DACS. This configuration relies on the relatively linear relationship between light scattering and exhaust particle mass concentration. A proportional-integral-derivative (PID) feedback control algorithm, programmed into the plenum nephelometer data acquisition module, achieves selected set-point concentrations in the chamber. The PID parameters were carefully tuned to yield approximately  $\pm 5\%$  mass concentration control through the variable diversion fan speed. The response time of this system is set between 30 and 60 s to allow some signal averaging and to dampen the short-term variation of the diluted exhaust concentration.

Near-continuous mass concentration measurements are taken in the chamber using a TEOM (Rupprecht & Patashnick Model 1400a, Thermo Electron Corporation, East Greenbush, NY) equipped with a PM<sub>2.5</sub> cyclone inlet. In tandem with the TEOM measurements, 37-mm Teflon filter samples were collected using Harvard Impactor samplers (Air Diagnostics and Engineering, Inc., Harrison, ME) equipped with PM<sub>2.5</sub> inlets, and operating by vacuum pump at 10 L/min.

TABLE 1Summary of TEOM-based PM2.5 mass concentrations(discrete 10-min averaging intervals) over 37 nominal 2-hmeasurement sessions

	Nominal concentration		
Parameter	$(\mu g/m^3)$ Filtered air	100	200
Number of runs	12	13	12
Average ( $\mu$ g/m <sup>3</sup> )	4.4	101.6	205.3
Standard deviation ( $\mu$ g/m <sup>3</sup> )	3	1.8	6.3
Coefficient of variation (%)	68.8	1.8	3.1
Minimum ( $\mu$ g/m <sup>3</sup> )	b.d.l.	98.2	194.2
Maximum ( $\mu$ g/m <sup>3</sup> )	9.0	104.0	212.2

### **EXPOSURE ATMOSPHERE CONCENTRATIONS**

The control feedback system maintains a stable concentration over the course of an exposure period, with a standard deviation within 2-h exposures of 2.9, 4.3, and 7.1  $\mu$ g/m<sup>3</sup> for nominal filtered air, 100, and 200  $\mu$ g/m<sup>3</sup> concentrations of PM<sub>2.5</sub>, respectively. Slight variation about the intended mass concentration (Table 1) is most likely due to small changes in the particle size distribution over time resulting in minor deviations from the fixed value of light scattering per unit mass assumed in the control-feedback program. Spatial uniformity of fine particle concentration within the exposure room was confirmed by concurrent placement of nephelometers adjacent to each other and then positioned at the front, center, and rear of the exposure room, followed by positioning along a potential left-middleright gradient. The real-time light scattering traces of the nephelometers were nearly identical  $(\pm 2.5\%)$  whether the instruments were adjacent or separated by up to 4 m inside the exposure room. Well-characterized mixing and plug flow conditions were previously documented by Wu et al. (1999) and Yost et al. (1999).



FIG. 2. Diesel exhaust particle source composition: carbon and trace elements.

The target  $PM_{2.5}$  mass concentration is repeatable from one measurement session to another, as summarized in Table 1. Table 1 shows the range and average concentration determined across multiple 2-h exposure sessions over a span of 9 mo.

The continuous TEOM measurements, averaged over the total exposure session duration, compare exceptionally well with the integrated PM<sub>2.5</sub> filter mass measurements. A comparison of TEOM versus Harvard Impactor measurements for 83 concurrent samples yields a slope of 1.01 and an  $R^2$  of .990. Multistage samples collected on a micro-orifice uniform deposition impactor (MOUDI) indicate a mass median diameter of 0.10  $\mu$ m ( $\sigma_g = 1.15$ ) determined for fine PM from 100- and 200- $\mu$ g/m<sup>3</sup> sample sessions. The total fine particle mass measured by the MOUDI compares favorably with the TEOM, yielding a slope of 0.98 and an  $R^2$  of .975 for eight concurrent samples.

Figure 2 summarizes the set of PM<sub>2.5</sub> species measurements taken in the exposure room. Elemental carbon (EC) and organic carbon (OC) levels were determined by thermal optical transmittance analysis of quartz filter samples, while metals concentrations were determined by x-ray fluorescence analysis of Teflon filter samples. Also shown is the diesel profile for comparable species abundances reported by EPA SPECIATE (U.S. EPA, 2007). The mass fraction determined from 27 samples ranging between 48 and 205  $\mu$ g/m<sup>3</sup> of PM<sub>2.5</sub> was 0.74 for EC and 0.23 for OC. The ratio of OC to EC among these samples averaged 0.34 ± 0.12, with a 95% CI of (0.21, 0.54). Our silicon mass fraction in excess of the SPECIATE level is partially explained by silicon mass in a filtered air sample that is higher than found in the method blank.

In addition to these particle data, the accompanying concentrations of nitrogen dioxide (NO<sub>2</sub>) measured with a Thermo model 42 chemiluminescent analyzer varied from 10 to 35 ppb for PM<sub>2.5</sub> levels of 50 and 200  $\mu$ g/m<sup>3</sup>, respectively, representing about 1.5% of the total NO<sub>x</sub> present. Due to the lack of exposure to sunlight, the NO<sub>x</sub> remains in a relatively reduced state, most of it in the form of nitric oxide (NO). Carbon monoxide levels measured with a Lear Seigler 9830 nondispersive infrared continuous CO analyzer averaged 0.30, 0.39, and 0.80 ppm for corresponding  $PM_{2.5}$  levels of filtered air, 100  $\mu$ g/m<sup>3</sup>, and 200  $\mu$ g/m<sup>3</sup>, respectively, measured across 41 exposures during a 9-mo interval. Aldehyde vapors were quantified using DNPH-coated Sep-Paks (Classic, Waters Chromatography Division, Millipore Corp.) operated at 0.5 L/min flow rate, subsequently extracted and analyzed by high-performance liquid chromatography (HPLC) with ultraviolet detection. Limited sampling revealed an average concentration (n = 5) at a nominal 100  $\mu$ g/m<sup>3</sup> PM<sub>2</sub> 5 level of 23.4  $\mu$ g/m<sup>3</sup> for formaldehyde, and 14.3  $\mu$ g/m<sup>3</sup> for acetaldehydes, the two most prevalent aldehyde compounds. The average ratio of acetaldehydes to formaldehyde in a set of 8 samples at PM<sub>2.5</sub> levels ranging from 3.0 to 202.  $\mu$ g/m<sup>3</sup> was 0.35, similar to the 0.34 ratio found in the EPA SPECIATE model light-duty diesel exhaust profile (U.S. EPA, 2007).

#### DISCUSSION

A diesel exposure facility has been assembled using a diesel engine under load and a two-stage exhaust dilution system with dynamic feedback control. The downstream exposure conditions are both stable and reproducible. While the conditions in the exposure system do not entirely replicate those in the atmosphere due to the relatively low ratio of NO<sub>2</sub> to total NO<sub>x</sub>, the fine particulate matter size distributions are quite similar to aged diesel exhaust a few hundred meters from a major roadway (Zhu et al., 2002). For inhalation toxicology purposes, the similarities of diluted PM to aged diesel exhaust are interesting as they permit us to separate effects of abundant DE components from NO<sub>2</sub>, the latter of which has a distinct toxicity profile.

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