

**2007 DIESEL PARTICULATE MEASUREMENT
RESEARCH**

**Project E-66
Executive Summary Report**

Prepared by:

Imad A. Khalek, Ph.D.

Prepared for:

**Coordinating Research Council, Inc.
U.S. Department of Energy/National Renewable Energy Laboratory
Engine Manufacturers Association
U.S. Environmental Protection Agency
California Air Resources Board**

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ENGINE, EMISSIONS AND VEHICLE RESEARCH DIVISION

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FOREWORD

Project E-66 was funded by the Coordinating Research Council (CRC), Department of Energy / National Renewable Energy Laboratory (DOE / NREL), US Environmental Protection Agency (EPA), Engine Manufacturers Association (EMA), and California Air Resources Board (CARB). The sponsors were represented by Mr. Brent Bailey and Dr. Chris Tennant from CRC, Dr. Doug Lawson from NREL, Dr. Bruce Cantrell and Mr. Matt Spears from EPA, Dr. Shirish Shimpi from Cummins, and Mr. Hector Maldonado from CARB.

The Southwest Research Institute (SwRI) Principal Investigator and Project Manager was Dr. Imad Khalek, Program Manager-R&D. Technical staff members who largely contributed to this work were Mr. Daniel Preece, Principal Technician, Mr. Joe Sosa, Senior Technician, Mr. Robert West, Staff Technician, Ms. Kathy Jack, Research Assistant, Mr. Keith Echtle, Laboratory Assistant Manager, and Mr. Ernest Kruger, Laboratory Manager.

The work was initiated and reviewed by the E-66 Panel members who are listed below in alphabetical order. Dr. Steve Cadle was the Chairman and Dr. Shirish Shimpi was the Co-Chairman of the E-66 Panel. Mr. Brent Bailey and Dr. Chris Tennant from CRC were the Project Managers representing the sponsors.

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ACRONYMS

COV	Coefficient of Variation
CPC	Condensation Particle Counter
CRT-DPF	Continuously Regenerative Technology-Diesel Particulate Filter
CVS	Constant Volume Sampler
DMM	Dekati Mass Monitor
DPF	Diesel Particulate Filter
EC	Elemental Carbon
EEPS	Engine Exhaust Particle Sizer
EMTC	Emission Measurement Testing Committee
ET	Exhaust Temperature
FFV	Filter Face Velocity
FTP	Federal Test Procedures
HDD	Heavy-Duty Diesel
NRTC	Nonroad Transient Cycle
OC	Organic Carbon
PDR	Primary Dilution Ratio
PEFB	Partial Exhaust Flow Bypass
PFSS	Partial Flow Sampling System
PM	Particulate Matter
PMP	Polymethylpentene
PP	Polypropylene
QA/QC	Quality Assurance/Quality Control
QCM	Quartz Crystal Micro-Balance
SDR	Secondary Dilution Ratio
SMPS	Scanning Mobility Particle Sizer
SPMS	Solid Particle Measurement System
SPR	Saturation Pressure Ratio
SRT	Secondary Residence Time
ST	Sampling Time
ULSD	Ultra-Low Sulfur Diesel
CARB	California Air Resources Board
CRC	Coordinating Research Council
DOE	Department of Energy
EMA	Engine Manufacturers Association
EPA	Environmental Protection Agency
ISO	International Organization for Standardization
NREL	National Renewable Energy Laboratory
US	United States
SwRI	Southwest Research Institute

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1.0 BACKGROUND

Since the promulgation of diesel exhaust particulate matter (PM) emissions standards in 1988, the United States (US) Environmental Protection Agency (EPA) has defined diesel exhaust particulate matter (PM) as the material that collects on a filter in a stream of exhaust that is cooled and diluted to a temperature of less than or equal to 52°C. Gravimetric analysis of the filter has been the basis for determining PM emissions from heavy-duty diesel (HDD) engines for certification and other research and testing activities.

Although PM emissions standards from on-highway HDD engines were reduced by more than 83 percent from 0.6 g/hp-hr in 1988 to 0.1 g/hp-hr in 1994, the established procedure for PM collection and quantification was considered to have been adequate for regulatory test procedural purposes.

In 2007, however, on-highway HDD engines are required to meet a PM emissions standard of 0.01 g/hp-hr which represents a 90 percent reduction from the 1994 level. In addition, the exhaust PM composition is expected to change because most engines will require catalyzed diesel particulate filters (DPFs) to meet the stringent PM emissions standard. PM composition is expected to consist mainly of volatile and semi-volatile hydrocarbon and sulfuric acid species derived from unburned and partially burned fuel and lubricating oil. This low level of volatile PM mass poses a technical challenge for the accurate mass measurement of PM using the pre-2007 established sampling protocol. Measuring a low quantity of PM mass deposited on a filter is a major challenge, but several other factors including filter handling, type of filter media, formation of artifacts, sampling system conditioning, and dilution parameter selection may also affect PM measurement.

Recognizing some of the PM measurement challenges for 2007, EPA modified the definition of PM by narrowing the filter face temperature from a peak of below or equal to 52°C to being continuously maintained in a range between 42°C and 52°C (47°C ± 5°C). EPA also implemented several changes to the secondary dilution tunnel of the constant volume sampler (CVS) as well as to the filter media, filter handling, and weighing chamber specifications in order to improve the quantification of PM mass emissions from engines that meet the 2007 standards [1]. CVS is the EPA-approved system for measuring HDD particulates.

Although the new PM measurement procedures specified by EPA for 2007 were demonstrated by EPA to achieve less than a 10 percent coefficient of variance (COV) at 0.004 g/hp-hr [2], preliminary data produced by SwRI for EPA, using the 2007 sampling procedures, gave a COV of 23 percent for four hot-start FTP transient tests at the 0.0034 g/hp-hr level [3]. Although the COV difference between SwRI and EPA may be partially explained by differences in the sampling and handling methodologies, background particle level, and the variety of engine and aftertreatment systems tested, these results suggest that additional effort is required to fully understand the variability present in PM measurement for engines equipped with DPFs.

While the new 2007 sampling methodology is expected to improve quantification of PM mass and reduce variability in comparison to the current sampling method, improvements were believed necessary and possible to achieve through further investigation. For example, several variables likely require more specific definition given the measurement challenges of 2007 PM emission limits. The objective of E-66 was to explore the benefit of more specific definitions of several variables on PM mass measurement. Parameter evaluations included:

- Secondary dilution tunnel geometry and residence time
- Filter face velocity
- Dilution air temperature
- Means of achieving the $47^{\circ}\text{C} \pm 5^{\circ}\text{C}$ filter face temperature
- Primary and secondary dilution ratio requirements
- Verification of filter material influence
- Filter equilibration time (currently time periods of 30 minutes to 60 hours are allowed)
- Primary and secondary dilution air filtration requirements
(Currently, 98% efficient HEPA filtration is required for primary dilution air,
and 99.97 % efficient filtration for secondary dilution air).

In light of these issues, Project E-66, entitled “2007 Diesel Particulate Measurement Research,” was initiated by the CRC Real World Vehicle Emissions and Emissions Modeling Group. The primary goal for this project was to investigate the above noted factors with the intention of improving future PM measurement. Additional investigations compared the PM emission performance of partial flow sampling system (PFSS) units and real-time PM measurement instruments to the CVS.

2.0 INTRODUCTION

Project E-66 consists of Final Reports on Phases 1 [4], 2 [5], and 3 [6] submitted to the project sponsors and is available to the public at the CRC Website (crcao.org). This executive report summarizes the main conclusions for all three phases of Project E-66. It also includes a set of recommendations for consideration in future activities requiring PM measurement from diesel engines.

3.0 OBJECTIVES

Project E-66 was divided into Phases 1, 2, and 3. The same diesel engine was used in all phases of the project. The engine was a 1998 DDC Series 60 HDD engine equipped with a continuously regenerative technology diesel particulate filter (CRT-DPF) or a CRT-DPF with a partial exhaust flow bypass (CRT-DPF with Bypass) to elevate PM emissions to near 70 to 80 percent of the 2007 PM emissions standard. The fuel used for Phases 1 and 2 was an ultra-low sulfur diesel (ULSD) fuel with a sulfur content of 6.9 ppm. Phase 3 ULSD fuel was from a different batch with a sulfur content of 3.8 ppm.

The objectives of Phase 1 were to:

- Minimize gas phase hydrocarbon adsorption on the filter used for PM collection from a dilute exhaust sample by using a carbon denuder upstream of the filter.
- Investigate the effect of filter media and filter face velocity on PM collection and emissions.
- Compare the performance of several real time particle measuring instruments including Engine Exhaust Particle Sizer (EEPS), Scanning Mobility Particle Sizer (SMPS), Dekati Mass Monitor (DMM), and Quartz Crystal Microbalance (QCM) with that of the CVS filter-based method that meets the 2007 PM sampling protocol.

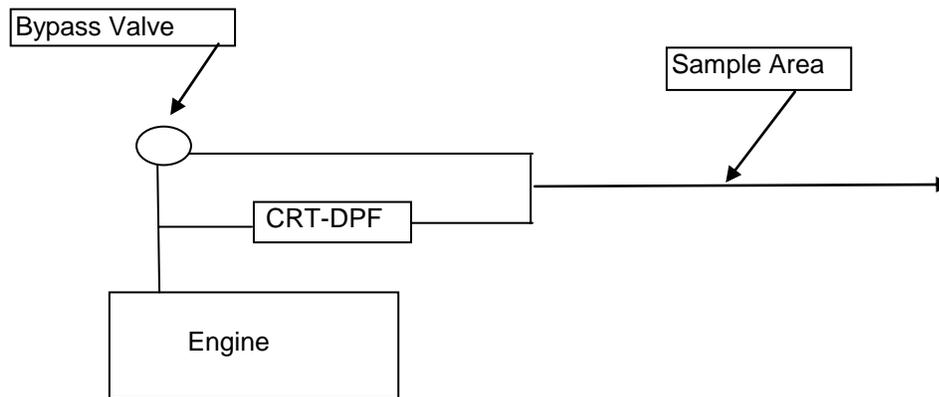
The objectives of Phase 2 were to:

- Investigate the effect of filter face velocity (FFV) and sampling time on solid and volatile particle collection by a Teflon[®] membrane filter, namely the Pall Teflo filter.
- Examine the effect of dilution conditions, such as the CVS primary dilution ratio, residence time and temperature, and secondary dilution ratio and residence time, on particle measurement using the real time particle instruments listed in Phase 1, and Teflo filters in limited experiments.
- Study the effect of exhaust stack and dilution system conditioning history on particle measurement.

The objectives of Phase 3 were to:

- Compare the PM emissions measured from the 1998 DDC Series 60 HDD engine equipped with a CRT-DPF using six different secondary dilution systems that were coupled to the full flow CVS. These dilution systems included one short and one long residence time secondary tunnels from SwRI that were used only with the full flow CVS, and four partial flow sampling system (PFSS) units, namely the Cummins AEI/CUM, AVL SPC, Horiba MDLT, and Sierra BG3. The PFSS units could be either used with the full flow CVS as secondary dilution systems, or used as independent dilution systems sampling directly from raw engine exhaust. Engine tests to generate exhaust species for these particulate measurement system evaluations consisted of two steady-state (rated speed, 100% and 10% load) and two transient engine tests, the Federal Test Procedure (FTP) and the Nonroad Transient Cycle (NRTC).

- Compare the PM emissions measured using all the PFSS units mentioned above on engine exhaust in addition to the Sensors micro-proportional sampler (MPS) with the PM emissions measured using the CVS. The work was performed using the CRT-DPF and CRT-DPF with Bypass, as illustrated in the following figure. The PM was collected on Teflo filters for all experiments. Quartz filters were used in very limited experiments using the CRT-DPF with Bypass. These filters were analyzed for PM organic and elemental carbon and sulfate using the Horiba MEXA-1370.



CRT-DPF BYPASS SYSTEM

4.0 SUMMARY OF MAIN FINDINGS

This summary report is intended to present the main findings for Phases 1, 2, and 3 of Project E-66. For more in-depth information, the reader is referred to Phases 1, 2, and 3 final reports, which are available at the CRC website.

4.1 Phase 1

This section includes the main findings from Phase 1 of Project E-66. Phase 1 investigated PM measurement artifacts and variability, filter face velocity, and real time particle instruments.

4.1.1 *Minimizing Gas Phase Artifact Collection*

Filters used for PM collection are prone to gas phase adsorption that add weight to the filter during PM collection resulting in “positive artifacts.” The gas phase materials are mainly volatile hydrocarbon species derived from partially burned and unburned fuel and lube oil. In Phase 1 of Project E-66, an attempt was made to add a carbon denuder in the PM sample train, upstream of the filter, to remove volatile hydrocarbon material from the gas phase before reaching the filter in order to reduce positive artifacts. The performance of the carbon denuder in adsorbing hydrocarbon was not consistent. Furthermore, the carbon denuder failed to regenerate after reaching saturation. Thus, it was decided early in the program to discontinue the development and use of the carbon denuder, and rely on the quality of filter medium itself to minimize gas phase adsorption.

4.1.2 *Effect of Filter Media on Quantification of PM Emissions*

Several filter media types were used on this project including:

Filter Media	Manufacturer	Initial Weight, mg	Material	Efficiency, ^d %	Thickness, μm	Pressure Drop, ^e inH ₂ O
Teflo	Pall	180	PTFE ^a Membrane, 2 μm pore size, PMP ^b ring	99.99	46/508 ^e	30
Teflon [®]	Whatman	143	PTFE Membrane, 2 μm pore size, PP ^c ring	99.70	40/ 380 ^e	90
PTFE-PP	Donaldson	157	PTFE Membrane, 2 μm pore size, PP ^c ring	99.99 ^f	40/508 ^e	78
PTFE-PE	Donaldson	119	PTFE Membrane, 2 μm pore size, PTFE ring	99.99 ^f	40/254 ^e	78
Zefluor	Pall	242	PTFE Membrane with PTFE support	99.99	152	60
TX-40	Pall	91	PTFE Coated Borosilicate Glass Fiber	99.90	178	30

^aPolytetrafluoroethylene (Teflon[®])
^bPolymethylpentene
^cPolypropylene
^dFollowing ASTM D 2986-95A 0.3 μm (DOP) at 32 L/min/100 cm² filter media.
^eTotal Ring Thickness
^f Approximate pressure drop at 75 cm/sec

For an engine equipped with a CRT-DPF, the reported PM emissions for tests conducted here ranged from 3% to 23% of the 2007 PM limit. These low PM measurements challenged the ability of filter weighing procedures that were specified for 2007, therefore, to improve the understanding of PM measurement capability at the specified 2007 limit, the E-66 investigators decided to bypass a portion of exhaust around the CRT-DPF to achieve a PM level that was about 70% of the 2007 limit, rather than the 3% to 23% range obtained with all exhaust passing through the CRT-DPF.

Performance of Teflon[®], Teflo, PTFE-PP, and PTFE-PE filter media was similar, i.e., no major PM difference was noted among these media. Performance of Pallflex TX-40 filters differed from the above media as follows: PM indicated by Pallflex TX-40 filters was greater than Teflon[®] and Teflo filters by a factor of approximately two when sampling CRT-DPF exhaust without Bypass at 5% of the 2007 PM limit, and by about 10% when sampling CRT-DPF exhaust with Bypass at 70% of the 2007 PM limit.

These tests showed that the Pall Teflo filter, a Teflon[®] membrane with a polymethylpentene ring, gave the lowest positive artifact mass and the lowest coefficient of variation in comparison to TX-40 and other Teflon[®] membrane filters. As a result of these experiments, a conclusion of E-66 was to recommend Teflo filters for future measurements of PM at the 2007 FTP PM level of 0.01 g/hp-hr.

4.1.3 Variability in PM Emissions Using CRT-DPF with and without Bypass

Forty-six repeat test cycles were generated for the hot-start FTP transient cycle using a CRT-DPF with an engine emission level at 5 percent of the 2007 PM standard (0.0005 g/hp-hr) and a coefficient of variation (COV) of 50 percent (0.00025 g/hp-hr). For the rated power condition, the COV was 35 percent based on 49 repeat test cycles. (These low level PM values could have been reported as 0.5 mg/hp-hr and 0.25 mg/hp-hr, but since regulations are stated in units of g/hp-hr, these units were retained although the large number of decimal places may appear awkward.)

For the CRT-DPF with Bypass at an emission level of 70 percent of the 2007 standard (0.007 g/hp-hr), the COV for the FTP was 8.6 percent (0.0006 g/hp-hr), a measurable improvement compared to the 50 percent COV without CRT-DPF exhaust Bypass.

If one assumes that the absolute values of the COVs obtained at 5 percent and 70 percent of the 2007 standard are total measurement errors that are independent of the PM emission level or the PM mass collected on the filter, one can predict the COV at the standard by dividing those absolute values over the 2007 PM standard of 0.01 g/hp-hr. Such calculations predict a COV in the range between 2.5 percent (0.00025/0.01 x 100) and 6 percent (0.0006/0.01x100) at the standard level of 0.01 g/hp-hr.

4.1.4 Effect of Filter Face Velocity on PM Emissions

For an engine equipped with a CRT-DPF with Bypass, the influence of filter face velocity at a constant 47°C filter face temperature is as follows:

<u>Engine Test</u>	<u>Filter Face Velocity Change</u>	<u>PM Decrease</u>
Hot-start FTP	24 cm/sec to 120 cm/sec	25%
Rated rpm, 10% load	60 cm/sec to 120 cm/sec	60%

These reported PM reductions are believed due to the volatile portion of PM as the following argument outlines, and as also confirmed by experiments performed during Phase 2 of E-66. Gas phase adsorption on the filter and filter saturation may be responsible for such large changes in measured PM emissions. For example, the flow volume passing through the filter at 120 cm/sec is about five times larger than the volume sampled at 24 cm/sec. If the filter that is running at 120 cm/sec reaches its adsorption capacity early during the FTP transient cycle, then its filter collected PM will not include positive artifact for the later portion of the cycle. In contrast, the 24 cm/sec filter reaches adsorption capacity toward the FTP cycle end, and is expected to result in a larger positive artifact.

Diesel particles are composites that consist of solid soot and adsorbed volatile species. The objective of the subject program is to collect these composite particles, to avoid accumulating gas phase volatiles onto the filter (positive artifact), and to avoid desorbing the volatile phase component of the composite particles (negative artifact). In order to minimize positive artifacts associated with gas phase adsorption on the filter during PM collection, it is best to reach filter saturation as soon as possible by going to a high filter face velocity. Also, higher filter face velocity seems to give a better agreement between the filter-based PM mass emissions and real time particle instruments that are insensitive to positive or negative artifacts. A filter face velocity that is too high (greater than 100 cm/sec) may result in a negative artifact.

4.1.5 Effect of Filter Face Temperature on PM Emissions

For the case where the engine was equipped with the CRT-DPF with Bypass set to provide a PM emission rate at 70 percent of the 2007 PM standard, results indicated no difference in measured PM emissions for the FTP transient cycle between using a Teflo filter face temperature of 47°C and 25°C. Studies in PM mass collection sensitivity to filter temperature were not performed at levels below 10% of the 2007 standard (Bypass disabled). Therefore, the aforementioned conclusions cannot be applied to a CRT-DPF without Bypass. Further study is required.

4.1.6 Filter Pre-Baking

To avoid contaminants loss from the sample filter during PM collection (negative artifacts) at a filter face temperature of 47°C ± 5°C, the Teflo filters were first pre-baked in a vacuum oven for 24 hours at a temperature of 52°C. After pre-baking and conditioning in the weighing chamber for 24 hours, each pre-baked Teflo filter lost about 5 µg of its original weight prior to baking. The 5 µg lost are likely to be some unknown volatile contaminant on the filter as received from the manufacturer.

In order to test whether or not pre-baking the filter is necessary for testing, PM emissions

from a total of 14 repeats of the FTP transient cycle were performed using pre-baked and unbaked Teflo filters. For an exhaust configuration using CRT-DPF with Bypass at an emission level of 70 percent of the 2007 PM standard (0.007 g/hp-hr) and a filter weight gain of about 70 µg, no significant difference was observed in PM emissions between using pre-baked and unbaked filters.

No experiments were performed to compare the PM emissions performance using baked and unbaked-filter for an exhaust configuration that includes CRT-DPF without bypass, where the PM emissions level for the FTP is expected to be at 5 percent of the 2007 PM standard (0.0005 g/hp-hr), with an expected filter weight gain of about 5 µg. Thus, it is not clear whether or not filter pre-baking will be necessary when testing with CRT-DPF without Bypass. Also, it is not clear whether or not different filter batches contain more or less contaminant than the one measured during this work. Thus, as a precaution, it is recommended that filter pre-baking be used to reduce filter batch to batch variability when using CRT-DPF without Bypass.

4.1.7 Real-Time Particle Instruments and the Filter-based Method

Three real time particulate instruments were compared with simultaneous measurements of CVS-based PM. These real time instruments were the DMM-230, EEPS and SMPS. The DMM-230 measures real time particle size and mass and the EEPS measures real time particle size and number concentration. The SMPS provides particle size and number concentration, but is limited to steady-state engine operation. If particle density is known, the EEPS and SMPS results can be converted to mass. A linear regression with a correlation coefficient (R^2) of better than 0.95 at the 95 percent confidence level was obtained for a total of 46 data points. Note that the observed good linear regression applies only to an exhaust configuration using a CRT-DPF with Bypass, where solid particles contribute to PM emissions. These results do not guarantee the same conclusion when using a CRT-DPF without Bypass, where the PM emission is composed mainly of volatile material.

Another real-time PM instrument, the Quartz Crystal Monitor (QCM) was tested, but results indicated that more in-depth investigation was required than the E-66 schedule permitted. In summary, proper operation of the QCM requires firm adhesion of all particles onto the quartz crystal, thus causing a unique instrument response. A unique response is not always attained for particles characterized by an agglomerate morphology. Particles that are attached to other particles, a typical agglomerate character that is true for diesel particulate, do not exert the same force on the quartz crystal as particles that firmly attach directly to the crystal. Under circumstances of agglomerates with very low particle deposit, for example, less than 0.5 micrograms, a QCM may demonstrate a unique response to mass deposit. Non-agglomerate particles may produce a unique response at higher than 0.5 microgram deposit.

4.1.8 Summary of Phase 1

- Minimizing gas phase adsorption on the filter by using a carbon denuder was not successful due to the difficulty associated with regenerating the denuder after saturation. Thus, the use of a carbon denuder was discontinued, and minimizing positive artifacts was achieved by the use of Teflon[®] membrane filters instead.
- Filter pre-baking in a vacuum oven at 52°C for 24 hours did not affect PM emissions, compared to unbaked filters using the CRT-DPF with Bypass. No experiments were done to verify the effect of pre-baking filters when using CRT-DPF without Bypass. Because the Teflo filter lost about 5 µg during pre-baking, an amount similar to filter weight gain for the FTP transient cycle when using CRT-DPF without Bypass, pre-baking is recommended as a precaution when using CRT-DPF without Bypass to minimize or avoid negative artifact during PM collection.
- At a PM emission level that is 5 percent of the 2007 standard, all Teflon[®] membrane filters such as those made by Pall, Whatman, and Donaldson reported equivalent PM emission levels at 0.0005 g/hp-hr, however, successive measurements could vary by approximately half of the mean value. In summary, a measured value is best reported as 0.0005 ± 0.00025 g/hp-hr. The Pallflex TX-40 filter, due to PM volatile artifact, measured a PM emission level that was a factor of 2 higher than that produced using the Teflon[®] membrane filters.
- At a PM emission level that was 70 percent of the 2007 PM standard, Teflon[®] filters measured 0.007 ± 0.0006 g/hp-hr. The PM emission level reported using a TX-40 filter was about 10 percent higher than the level reported using the Teflon[®] membrane filters, likely due to volatile artifact. This artifact (10% of 0.007) was slightly larger than the Teflon[®] membrane filter 1s (±0.0006). The Pall Teflo filter gave the lowest variability (±0.00038 g/hp-hr) along with the lowest pressure drop among all Teflon[®] membrane filters tested. This filter was selected for subsequent E-66 sampling from engines with wall-flow DPFs.
- PM measurement coefficient of variation (COV) summary:
 - At <10 percent of the 2007 PM standard, COV is 50%.
 - At the 2007 PM standard, COV is expected to be 6%.
- Increasing filter face velocity from 24 cm/sec to 120 cm/sec produced a 25 percent reduction in measured PM emissions during the FTP transient cycle. Controlling filter face velocity to a narrow range may help reduce the variability in PM measurement.
- A linear regression with a correlation coefficient (R^2) of better than 0.95 was obtained for n=46 tests between the CVS filter-based PM mass and PM mass derived from real time instruments such as the DMM-230, EEPS, and SMPS, using CRT-DPF with Bypass. Real time instrument vs. CVS correlation was not evaluated when using CRT-DPF without Bypass.

4.2 Phase 2

Phase 2 was performed using the same Phase 1 engine equipped with a CRT-DPF. Teflo membrane filters were used throughout the entire project phase as the preferred filter medium unless otherwise specified. Phase 2 investigated filtration efficiency and sampling time when using Teflo filters. Phase 2 also investigated the effect of dilution ratio and residence time using real time instruments, and secondary dilution residence time using Teflo filters.

4.2.1 Particle Mass Filtration Efficiency of Teflo Filters

Unlike gaseous emissions that can be reported as a single number, usually concentration, particle measurement consists of a two-dimensional array involving size and concentration. The following brief table reports Teflo filter particle mass filtration efficiency and particle size ranges for 60 to 129 cm/sec filter face velocities. These experiments were performed using diesel exhaust treated to remove volatiles and thus provide only solid particles to the Teflo filters.

<u>Particle Size</u>	<u>Mass Collection Efficiency</u>
7nm to 300nm	99%
<30nm	95%
<10nm	85%

This work showed that the filtration efficiency of solid exhaust PM mass is high, over 99 percent within the range of 60 to 129 cm/sec filter face velocity. It also demonstrated that the effect of filter face velocity on PM mass collection observed in Phase 1 was mainly due to volatile PM and not solid PM.

4.2.2 Effect of Sampling Time on PM emissions

At rated speed, 100 percent load, changing sampling times and face velocities resulted in the following observations:

<u>Sample Time Increase</u>	<u>Filter Face Velocity</u>	<u>Decrease in PM Reported</u>
20 min. to 50 min.	125 cm/sec	40%
60 min. to 150 min.	30 cm/sec	70%

These results are for an exhaust aerosol that was about 5 percent of the 2007 PM emission standard. It is important to note that the effects of filter face velocity and sample time on filter-based PM emissions are not independent of each other. High filter face velocity and short sample time may be equivalent to low filter face velocity and long sample time only if the flow volume is the same, and particle phase volatile material depositing on the filter is stable. In case the aerosol is unstable and subject to evaporation, the long sample time may bias the results to lower PM emissions due to negative artifacts.

In order to reach filter gas phase saturation as quickly as possible, a high filter face velocity is desired. In order to minimize negative artifacts, a short sample time is desired, but in order to minimize positive artifact a long sample time is desired. However, in most engine testing, the sample time is dictated by the length of the cycle to be run. Thus, the only means of minimizing positive artifact is by going to a high filter face velocity within the upper limit defined by EPA at 100 cm/sec.

In cases where the length of testing is not predefined, one tends to increase filter face velocity and sample time to maximize PM loading on the filter, particularly when the PM emission level is well below the 2007 PM standard. While increasing filter face velocity and sample time minimizes positive artifact collection relative to the total particle mass, it may lead to negative artifacts; this practice presents a challenge for filter media to accurately evaluate PM emissions at an emission level well below the 2007 PM standard.

4.2.3 Effect of Dilution Ratio, Residence Time and System History on PM Emissions Using Real Time Particle Instruments

The SMPS, EEPS, DMM-230, and QCM were used to study the effect of dilution parameters on PM emissions. With some of these real time instruments, a large matrix of different dilution parameters such as primary and secondary CVS dilution ratio and residence times were investigated. Teflo filters were only used later to determine the effect of secondary residence time on PM emissions for comparison with that observed using real time instruments.

Depending upon the engine operating condition, increasing the primary dilution ratio from 4 to 9 while holding the secondary dilution ratio at two resulted in 5 to 20 fold increase in PM emissions using SMPS, EEPS, DMM-230 and QCM. Since these four instruments employed three different physical measurement principles, some differences in particle mass were expected, however, all of these instruments reported similar response to dilution ratio, namely, higher dilution ratio increased the indicated PM. Increasing the secondary dilution ratio from 1.5 to 4.5 while holding the primary dilution ratio at two resulted in two-fold PM mass increase. Primary dilution ratio changes have greater influence on particle mass than secondary dilution ratio changes, within the range of dilution ratios investigated here, thus lab to lab comparisons at 2007 particle emission levels need to coordinate primary dilution ratios more carefully than secondary dilution ratios, but the factor of two influence of secondary dilution ratio is not to be viewed as unimportant.

The secondary dilution residence time had a very significant influence on particle mass emissions. A long residence time of 10 to 30 seconds showed an increase in PM mass emissions by more than two orders of magnitude, compared to a residence time of less than one second, using data obtained from the real time particle instruments. During Phase 3, this phenomenon was also demonstrated using Teflo filters, but the increase in PM emissions was a factor of three. This was likely due to the use of a different ULSD fuel in this part of the study with about half the sulfur content and 1/10th the aromatic content of the fuel originally used with the real time instruments.

Results during E-66 indicated that engine history, DPF, and dilution systems influenced the reported particle mass emissions from engines due to storage and release phenomenon of volatile and semi-volatile exhaust species. These observations have also been reported by other investigators, and indicate the importance of adhering to clearly defined testing protocol to ensure test to test and lab to lab repeatability.

The use of real time instrumentation in this phase of E-66 provided insight into diluted exhaust particle formation sensitivity. This limited study suggests that the details of dilution air conditioning and dilution system dynamics specific to mixing and mixture aging may have effects of varying significance on PM measured, regardless of the measurement technique applied (real time or gravimetric). In this study, it was observed that PM emissions from one

DPF-equipped engine were measured to be as low as 5 percent of the 2007 PM standard or as high as the standard, depending on the dilution parameter combination and real time instrument selected, however, the magnitude of this sensitivity is likely to vary between engines and aftertreatment systems. It is believed that a strong interdependence exists between exhaust constituency (sulfate and organics content in combination with remaining soot), dilution mixing characteristics (including residence time) and dilution air properties, including temperature and humidity. It must be recognized that a complete understanding of the mechanics of particle formation and growth under real-life dilution system conditions is well beyond the scope of E-66 and does not as yet exist.

4.2.4 Summary of Phase 2

- The change in filter face velocity did not have much influence on solid particle filtration efficiency using a Teflo filter. The filtration efficiency remained over 99 percent for filter face velocities between 60 cm/sec and 130 cm/sec. Thus, the influence of filter face velocity on particle mass collection that was observed during Phase 1 is mainly due to the presence of volatile PM.
- Longer PM sampling time led to a reduction in the reported PM emissions. Increasing the PM sampling time reduces the contribution of positive artifact collected by the filter relative to the total PM mass collected, but also contributes to the negative artifact evaporation loss of volatile PM material collected by the filter. Thus, long sampling time is not recommended unless the advantages of avoiding positive artifact outweigh the disadvantages of negative artifact.
- The influence of primary and secondary dilution ratio on particle mass measurement was demonstrated using the real time instruments. The primary dilution ratio had a strong influence on particle emissions. Changing the primary dilution ratio from 4 to 9 resulted in a factor of 5 to 20 PM mass increase. Changing the secondary dilution ratio did not have as much influence as changing the primary dilution ratio using a CRT-DPF without bypass.
- Increasing the secondary dilution residence time from the usual <1 second to a range of 10 to 30 seconds increased the reported particle mass emissions by two orders of magnitude, as determined by the real time instruments. The influence of secondary residence time was also demonstrated using Teflo filters, but only a particle mass increase by a factor of three was observed. Further investigation of reasons for the observed order of magnitude vs. factor of three difference is recommended.

4.3 Phase 3

In light of the EPA 2007 PM sampling protocol and the improvement made by the PFSS manufacturers relative to response time, the CRC Real World Vehicle Emissions and Emissions Modeling Group initiated the work under Phase 3 of Project E-66 to investigate the performance of PFSS compared to the full flow CVS using the 2007 PM sampling protocol with a diesel engine that meets the 2007 PM standard.

The main objective of Phase 3 was to evaluate several different partial flow sampling systems (PFSSs) used as either secondary dilution systems coupled to the full flow CVS, or as stand-alone systems to sample directly from an exhaust pipe and perform the entire task of exhaust dilution. The logic of implementing Phase 3 as the last phase of Project E-66 was to conduct the experiments in the best possible way using recommendations and practices learned in Phases 1 and 2 of the project.

Phase 3 was performed using CRT-DPF and CRT-DPF with Bypass. Although some results of Phase 3 may be compared with those of Phases 1 and 2, note the following fuel differences. Compared to fuel of Phases 1 and 2, the Phase 3 fuel had a factor of 10 lower aromatic compounds, and a factor of 1.8 lower sulfur. The Phase 3 fuel differed since the Phase 1 and 2 fuels had been depleted.

4.3.1 *PFSS Response Time*

The response time of all PFSS units was better than 200 milliseconds with excellent proportionality to exhaust flow changes during transient operations. Evaluation of previous PFSS versions concluded that response time performance required major improvements because PFSS sample flow did not correspond to changes in exhaust flow, thus violating proportional sampling requirements of a PFSS. In contrast, the present versions of BG3, SPC, MDLT, AEI/CUM and MPS demonstrated excellent proportionality between exhaust flow and sample flow. The correlation coefficient of exhaust to sample flow was 99 percent or better for the BG3, AEI/CUM, MDLT, and SPC; and better than 97 percent for the MPS. The standard error relative to the average was generally below five percent, except for the MPS where the standard error was about 7 percent for the FTP and 13 percent for the NRTC. It is important to note that the correlation was based on all data points collected; however, CFR Part 1065 allows for the removal of five percent of the data collected for this correlation which would improve the correlation coefficient and standard error reported above for all the PFSS units. The exhaust flow sampling proportionality performance of these PFSS units complied with the CFR Part 1065 requirements that were in place during this investigation (Part 1065 is not final as of this publication date).

4.3.2 *PFSS as Secondary CVS Dilution Systems*

The BG3, SPC, MDLT, and AEI/CUM, when used as secondary dilution systems on the full flow CVS, measured similar average PM emission levels as the CVS (near 0.0005 g/hp-hr) with mean values that differed from each other within the CVS repeatability on the FTP (± 0.00025 g/hp-hr). The CVS repeatability of 0.00025 g/hp-hr and differences among PFSS of 0.00025 appear to be high. However, note that the PM emissions comparison was performed at PM emission levels below 10 percent of the 2007 standard. Also note that this comparison was with an exhaust configuration that did not include the CRT-DPF Bypass. Furthermore, the PM

emission levels obtained were approximately the same magnitude as the PM level obtained using a tunnel blank filter without engine operation. The very low PM level at or below 10 percent of the 2007 PM standard presents a laboratory challenge. Although all of today's best laboratory practices were performed, the similarity of tunnel blanks and engine results is evidence of that challenge and suggests that additional effort may be appropriate to refine present laboratory procedures. This is presently being investigated by EPA and engine manufacturers.

4.3.3 PFSS Measuring CRT-DPF Emitted PM Directly from Exhaust

The BG3, SPC, MDLT, and AEI/CUM, when used as partial flow systems measured similar average PM emission levels with mean values (near 0.0006 g/hp-hr) that differed from each other within the CVS repeatability on the FTP (+/- 0.00025 g/hp-hr). The CVS repeatability of 0.00025 g/hp-hr and differences among PFSS at 0.00025 appear to be high, however, as noted for these comparisons as secondary dilution systems, the mean values were very low, only 6% of the 2007 PM limit. The AEI/CUM reported PM emissions were 50 to 70 percent lower than those reported by the CVS. The MPS reported PM emissions about four times higher than a CVS filter measurement. For the AEI/CUM, it is likely that using 47°C dilution air temperature resulted in lower PM collection on the filter, compared to the CVS method, which used a secondary dilution air temperature of about 27°C. For the MPS, it was not clear why the reported PM emission level was higher than the CVS method. It is noteworthy that these results were measured by the CVS at 0.0006 g/hp-hr, which was 6% of the required 2007 PM limit of 0.01 g/hp-hr.

4.3.4 PFSS with CRT-DPF with Bypass

Three observations or factors suggested that E-66 should consider testing at 70% rather than 6% of the 2007 PM regulated limit. These were:

- +400% to -70% variation of PFSS from CVS when using direct sampling of CRT-DPF exhaust at only 6% of the 2007 regulated PM limit
- The same magnitude results for CVS-sampled engine data and tunnel blanks observed in Phase 1 when measuring exhaust at 6% of the 2007 PM limit
- The potential for actual PM levels at 70% of the 2007 PM limit that could be observed when 2010 NO_x aftertreatment is required.

The 70% PM limit testing was achieved with a bypass loop around the CRT-DPF. As noted above, this hardware configuration was termed CRT-DPF with bypass. PFSS performance when sampling the bypass exhaust mix resulted in improved ability to measure PM. For example, except for one PFSS that later evaluation resolved, most of the PFSS for steady-state engine operation demonstrated improved performance characterized by agreement within +/-30% of the CVS, a major improvement compared to the previously noted +400% to -70% differences. Results for transient testing were even better, with differences between CVS and PFSS ranging from 1% to 30% with one exception. These results indicated that improvement was achieved and suggested that attempting to measure filter-based PM emissions at <10% of the 2007 limit is a significant challenge.

As noted above, PFSS performance improvement resulted when exhaust PM concentration was increased through the use of a bypass. Opportunity remains for further improvement through examination of other variables related to particle physics. For example,

additional work with the SPC showed that when the residence time, dilution ratio, and dilution air temperature were matched with that of the CVS, the difference in PM emission results at rated speed, 100 percent load, narrowed significantly. Thus, future work should focus on comparing the PFSS with the CVS under very tightly defined dilution parameters. However, there should also be a recognition that the dilution processes of CVS and PFSS are different, and they may not agree under all engine exhaust conditions or with changes in engine technology. At present, EPA requires verification of equivalence between CVS and PFSS for transient cycle-based certification.

4.3.5 PFSS with Quartz Filters

Relative to the work with quartz filters using CRT-DPF with Bypass, the results were qualitative in nature due to the low level of PM collection on the filter and the possible elemental carbon (EC) artifact formation by pyrolysis during the OC portion of the PM analysis using the MEXA 1370 PM. When using the MEXA 1370 for quartz filter analysis, a stream of nitrogen is first introduced over the filter at a temperature of 980°C to desorb all OC material deposited on the filter. During this process, some of the OC may decompose and form EC on the filter. Typically, an optical method is used to correct for the EC artifact, but, in the case of MEXA-1370-PM no artifact correction is implemented. Thus, some of the EC usually reported using this method may be an artifact.

The AVL SPC showed higher emissions at rated engine power, not only relative to EC, but also relative to organic carbon (OC) and sulfate. The observed results particularly relative to the increase in OC and sulfate support the data obtained when the dilution parameters between the SPC and CVS were matched. The EC results, however, do not support the agreement obtained between the CVS and the SPC as a result of changing the dilution parameters, suggesting a potential EC artifact due to the method used for PM analysis or some other unknown behavior with the instrument that is not well understood.

The Sensors MPS seemed to underestimate EC in comparison to all other systems. This indicates that particle losses could be a problem with this system, or the analytical technique used for EC analysis is not accurate due to the very low level of EC collected with the MPS. At any rate, it will be useful to conduct a particle loss experiment with the MPS to better understand particle losses in the sample train.

The AEI/CUM that used a 47°C dilution air temperature seemed to minimize the collection of sulfate on the filter. This temperature may play a role in reducing the potential nucleation and growth of particles because the diluted exhaust never sees a temperature below 47°C during the dilution and cooling of the raw exhaust stream. Hence, using 47°C instead of 25°C dilution air temperature may suppress particle formation or condensation/adsorption during the dilution process, resulting in less PM mass collection by the filter. It is suspected that particle nucleation and growth and evaporation may not be totally reversible and may have some amount of hysteresis that inadvertently prevents material from going back into the gas phase. Thus, using a dilution air temperature of 47°C ± 5°C and maintaining a filter face temperature of 47°C ± 5°C, as was practiced in the AEI/CUM PFSS, seemed to result in less volatile hydrocarbon and sulfate PM deposit on the filter, compared to a dilution air temperature of 25°C ± 5°C and a filter face temperature of 47°C ± 5°C, which is the currently adapted and recommended practice.

4.3.6 Summary of Phase 3

The main objective of Phase 3 was to evaluate several different partial flow sampling systems (PFSSs) used as either secondary dilution systems coupled to the full flow CVS, or as stand-alone systems to sample directly from an exhaust pipe and perform the entire task of exhaust dilution.

- PFSS (BG3, SPC, MDLT, AEI and MPS) evaluated in Project E-66 demonstrated their ability to follow a transient test with better than 200 ms response, and with 97% to 99% proportionality between exhaust flow and sample flow, and demonstrating compliance with 40 CFR Part 1065 as described at the time of these tests.
- As the secondary dilution system on the full flow CVS, these PFSS (BG3, SPC, MDLT and AEI) yielded similar PM results to a system consisting of a full flow CVS and a secondary dilution system.
- When applied as PFSS sampling directly from an exhaust pipe that delivered PM at 6% of the 2007 regulated limit, several of these systems were equivalent to a CVS within measurement variability, while two others ranged from 400% high to 70% low.
- When PM levels were 70 percent of the 2007 PM standard, with one exception, these PFSS units improved from the +400% to -70% deviation from CVS (when measuring at 6% of the PM limit) to +/- 30% deviation from a CVS.
- Additional studies with quartz filters and other studies with dilution ratio and dilution air temperature variations explained some of the measurement differences among PFSS units.

5.0 RECOMMENDATIONS

5.1 Filter Media and Handling

The recommendations in this section include discoveries made during Project E-66 and SwRI laboratory practices known to be effective for HDD particle measurement.

- Teflo membrane filters are recommended for PM emission measurement due to their low affinity for artifact formation. In case chemical analysis of the PM sample is required, it is recommended that the feasibility of using the Donaldson-manufactured Teflon[®] membrane filter with the Teflon[®] ring be explored because of the inert nature of the ring support.
- Performing at least three consecutive filter weights before and after testing best determines an average weight gain. This practice was performed throughout Project E-66 and was very beneficial in obtaining a robust filter weight before and after testing.
- To prevent an electrostatic charge buildup, it is recommended that filters be placed on a pair of polonium 210 radioactive strips for at least 30 seconds before weighing and tweezers used to handle filters must be grounded.
- Condition clean filters for at least 24 hours in the weighing chamber before first weighing. After 24 hours of conditioning a stable filter weight was obtained with various filter media tested under Project E-66.
- Project E-66 did not show a difference in PM emission performance between a pre-baked and unbaked Teflo filter media, using the CRT-DPF with Bypass. Thus, it is recommended that filter pre-baking in a vacuum oven for 24 hours at 52°C becomes optional for CRT-DPF with Bypass or similar configurations of engine-out particle concentration. It is still recommended that filters be baked when using a DPF without Bypass. The Teflo filter typically loses about 7 micrograms after pre-baking for 24 hours. Different filters obtained from different lots may yield different responses to the filter baking protocol.

These results suggest that current test and filter handling protocol for PM mass measurement is challenging, and may be replaced in the future if an alternative is identified. The use of filters for PM collection using current methods is not practical, particularly when the PM emission level is well below the 2007 PM standard. Attempting to overcome a low filter weight challenge by using artificially low overall dilution ratio is not recommended. E-66 testing indicated that dilution ratio exerted a measurable influence on the PM reported; therefore forcing a test at low dilution to achieve a minimum net filter mass to improve weighing accuracy may distort the overall result.

5.2 Dilution Parameters and Filter Face Velocity

To reduce variability of PM mass measurement, it is recommended that PM sampling protocol in 40 CFR Part 1065 be reexamined. The following parameters must be narrowly defined in order to produce consistent and repeatable results:

1. Filter Face Velocity
2. Secondary dilution residence time
3. Secondary dilution ratio
4. Secondary dilution air temperature
5. CVS primary dilution ratio
6. CVS primary dilution residence time
7. CVS primary and secondary tunnel dilution air temperature.

Discussions on tightening some of these parameters are already underway as a part of EMA's Emission Measurement and Testing Committee (EMTC) that is composed of EMA, EPA and CARB members and includes other stakeholders such as testing facilities and instrument manufacturers. Based on the latest EMTC discussions, the following modifications have been proposed for Part 1065. They apply to both partial flow sampling systems (PFSSs) and CVS, unless otherwise specified.

- a. Maximize the filter face velocity as much as practically possible but not to exceed 100 cm/sec.
- b. Set the secondary dilution residence time to at least 0.5 sec but not to exceed 5 seconds (not applicable to PFSS).
- c. Set the total dilution residence time to at least 1 second but not to exceed 5 seconds.
- d. Set the primary dilution ratio to at least 2 at the maximum exhaust flow rate expected during a transient cycle.
- e. Set the total dilution ratio to a value between 5 and 7 at the maximum exhaust flow rate expected during a transient cycle.
- f. Set the dilution air temperature to 25°C +/- 5°C anywhere upstream of the mixing point, but the closer the better.
- g. Set the transfer tube length between the exhaust pipe internal wall and the point of mixing to no more than 26 cm (applicable only to PFSS).

5.3 Real Time Particle Instruments

Results of E-66 suggest that a steering committee be established to design, verify, and implement a standard operating protocol for real time particle instruments that may be used as substitutes for the filter-based method or may be used for onboard PM measurement. Some of the needs are to:

- Evaluate particle loss within instruments and sampling systems
- Define zero, span and linearity check for different concentration levels
- Define instrument accuracy determination
- Define a calibration protocol
- Specify calibration particle material
- Define an instrument performance comparison method; e.g., compare to filters or some other accepted method
- Define a standard operating procedure.

5.4 Partial Flow Sampling Systems

Project E-66 evaluated PFSSs at a time when production versions of most were available, except for the MPS, which was a prototype. The response time and mechanical operation of the production versions are capable of correctly determining a proportional sample mass flow rate on a real time basis from measured exhaust mass flow values produced during USEPA on- and off-road diesel engine transient cycles, extracting said proportional exhaust sample from the exhaust stack and diluting the raw sample for gravimetric PM measurement as defined in 40CFR Part 1065. All remaining correlation issues are related to PM emissions differences between PFSSs, or between PFSSs and CVS or between CVS systems. However, with the proposed tightening of dilution parameters and filter face velocity being considered by EMA, EPA, CARB and other stakeholders in the Emission Measurement and Testing Committee (EMTC), the differences may be reduced to an acceptable level within and among laboratories.

6.0 REFERENCES

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