The DAAAC Protocol™ for Diesel Aftertreatment System Accelerated Aging

Eakle, S.T. and Bartley, G.J. (2014)
The DAAAC Protocol™ for Diesel Aftertreatment System Accelerated Aging.
In "Proceedings: Emissions 2014."
Emissions 2014 Conference 11-12 June 2014, Troy, Michigan, USA.
M. Nasim Uddin, editor.
ISBN: 978-1-63439-002-6

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Abstract

Accelerated aging of automotive gasoline emissions catalysts has been performed on bench engines for decades. The EPA regulations include an accelerated aging cycle called the Standard Bench Cycle (SBC) that is modeled on the RAT-A cycle developed by GM Corp. and published in 1988. However, this cycle cannot be used for diesel aftertreatment components because it is based on stoichiometric operation, whereas diesel engines typically operate under excess air (lean) conditions.

The need for accelerated aging cycles for diesel emissions systems can be illustrated by considering that the full useful life requirement in the United States for an on-highway truck is 435,000 miles, and an off-road application may be 8,000 hours. Aging under normal operating conditions is time-consuming and expensive. This need was recognized, and the DAAAC Protocol™ developed to provide accelerated aging cycles for the vast majority of diesel emissions system applications.

This paper describes the DAAAC Protocol™ in detail for the first time. The Protocol has been used for the EU-VI legislation for qualifying replacement emissions systems, and may be considered in the US for future regulations and emission system certification.

Introduction

Heavy-duty diesel engines come in many sizes and are used in a wide variety of applications. They may be used in mobile applications, on-road such as trucks and buses, or off-road such as construction equipment. They may also be used in stationary applications such as for power generation. The multiple applications require significant consideration of the emissions approach and equipment used. Aftertreatment components that are employed in many of these systems are primarily used for Particulate Matter (PM) and NO_x abatement.

For on-road applications, the emissions systems must meet Federal and/or State durability requirements. For example, a Class 8 truck may be required to demonstrate emissions compliance to 435,000 miles of operation. However, for practical and commercial purposes the equipment will ideally have at least one million miles of durability. A locomotive engine may require several years of 90 percent operation, and off-road equipment may require durability of several thousands of hours of operation. Developing the aftertreatment systems for all of these applications requires in-house assessment of the candidate systems durability, as well as any certification requirements. Clearly, it is not practical to develop emissions systems by running them under normal application conditions as it takes too long and is extremely expensive.

The DAAAC Protocol™ (The Protocol) is a diesel aftertreatment accelerated aging procedure that results in catalytic system deterioration similar to that observed in field-aged components. The procedures that were developed aimed to shorten durability testing times dramatically, with a reduction in durability testing time goal of ≥ 90 percent. Significantly shorter aging times markedly reduce the costs of durability testing. With today's rising energy prices, a large amount of any durability testing project can be spent simply burning fuel.

One aspect of diesel aftertreatment aging is thermal aging. The effects of high temperature exposure on catalyst performance have been well documented, and are a prominent part of the standard bench cycle (SBC) used for gasoline vehicle certification. In this case, use of elevated temperature for the aging cycles, and the Arrhenius rate law, shown in Equation (1), are applied in the Protocol.

activation energy

rate constant

$$k = Ae^{-\frac{E_A}{RT}} \qquad (1)$$

kelvin temperature

the gas consant

frequency factor or pre-exponential factor

mathematical quantity, e

Unlike the accelerated aging procedures that are used in gasoline applications, chemical aging (primarily from lube oil exposure) cannot be simply ignored. As stated previously, diesel aftertreatment systems have significantly higher durability requirements, hence a longer exposure to lube oil constituents. For example, ash accumulation (primarily from lubricating oil) within the DPF is a concern. Ash cannot pass through the walls of a wall-flow DPF, and accumulates in the channels. Ash can also contribute to increased pressure drop across the DPF, resulting in higher engine backpressure, and higher localized temperatures within the DPF during PM regeneration. Also, phosphorus from lubricating oils is known to adversely affect the activity of the oxidation catalyst in a catalyzed DPF, reducing passive regeneration performance [1]. For these reasons, the Protocol includes the chemical aging affect on the catalytic components due to the extent of lube oil exposure.

This paper presents the latest version of the DAAAC Protocol. The Protocol covers field data collection, field data processing, preliminary DAAAC aging cycle definition, bench engine setup, preliminary aging cycle operation with data collection, bench engine data processing and final DAAAC aging cycle definition. Emissions performance tests and sound physical analyses were used to correlate field-aged and bench-aged (using the DAAAC Protocol) aftertreatment systems. Full bench-engine emissions tests were performed on a degreened, field-aged and bench-aged diesel aftertreatment system. Subsequently, various physical analyses were performed on the catalysts to compare the characteristics of the field- and bench-aged catalysts, and to better understand the performance test results. The combined data demonstrated good correlation between the field- and bench-aged catalysts.

DAAAC Protocol™

The DAAAC program objective was to develop an engine bench accelerated aging procedure for diesel aftertreatment systems. This procedure has been formalized in the DAAAC Protocol illustrated in Figure 1. The Protocol requires that a limited amount of field data be generated using the intended application equipment. The various parts of the data are then processed according to the Protocol to derive the conditions for the application-specific DAAAC aging procedure.

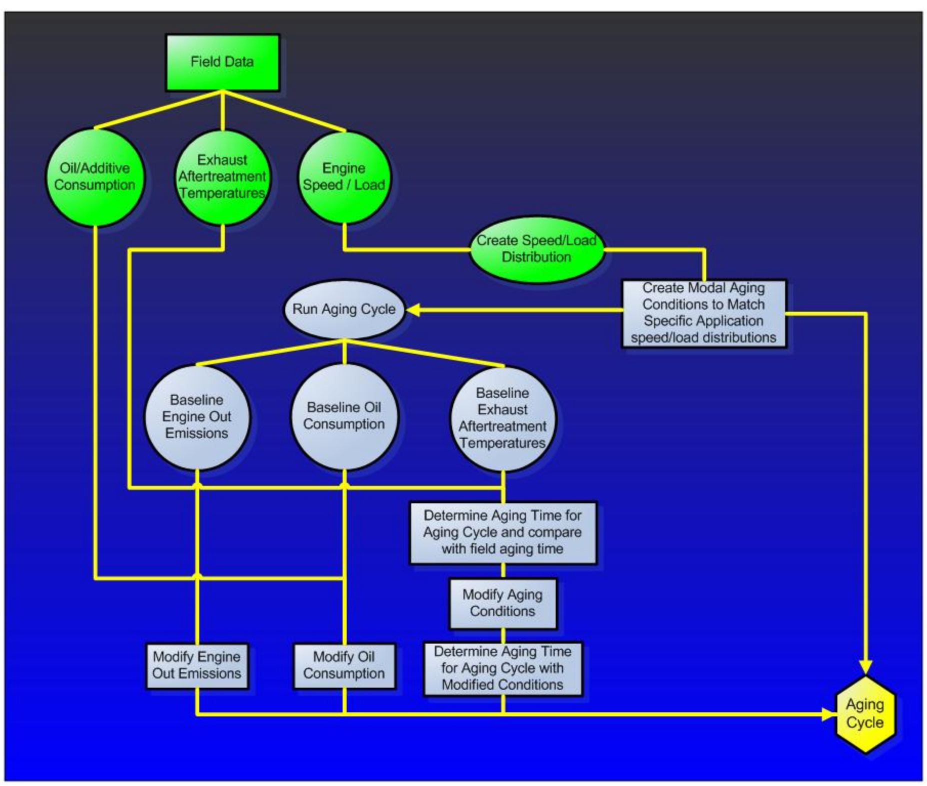

Fig. 1: The DAAAC Protocol Flowchart

Field Data Collection

The first step in the Protocol is to run the application under typical field conditions. Here, typical field conditions are defined as the application engine with the intended aftertreatment system installed in its intended configuration, and the full engine/aftertreatment control setup. The engine is to be operated as it will be operated in the real application. Ideally, this field work will not have to run for more than 10 percent of the full useful life field operation. It may run considerably less as long as all of the relevant data are obtained. To apply the Protocol, the following data should be collected:

Modal engine speed and load (normalized)

Engine speed and load data should be collected. It is assumed that engine load will already be normalized. Therefore no further post processing is needed. Engine speed will need to be normalized over the range of idle to high idle engine speed for the given application. For example, idle = 0%, high idle = 100%.

Modal exhaust gas temperatures

Accurate determination of accelerated thermal aging conditions requires a realistic and complete set of field temperature data. Temperature excursions that may result in significant thermal deterioration of a catalyst can occur in a few seconds, and such temperature excursions should not be missed by the data collection. Catalyst inlet temperatures (exhaust gas temperature upstream of a catalyst) may be used, but catalyst bed temperatures (gas temperatures within the catalyst matrix) will provide more ideal data. Many catalytic reactions are exothermic (heat generated) leading to localized temperatures within the catalyst that are higher than the inlet temperatures. These higher temperatures deactivate the catalyst more rapidly, and are the ideal temperatures to recreate in the bench engine aging cycle.

Total Operating Distance and Time

For applications involving recordable distance traveled, the total distance traveled over the data collection period should be recorded. The total engine operation time must be recorded for all applications. Here, engine operation time is defined as the time when the engine is running, and should not include any time when the ignition is on but the engine not running.

Lubricating Oil Formulation and Oil Consumption

The Protocol requires that the oil used during field data collection also be used for all of the accelerated aging work. The properties of the oil should be recorded, but are not required as inputs to the Protocol as long as the field and aging oil are the same.

The total oil consumed by the engine during the data collection period should be recorded. Any suitable method may be used to determine the total oil consumed. Records should be kept of oil additions (top offs) during this period. The oil change interval should be clearly stated and adhered to during the data collection period. The oil change interval will be used in setting up the accelerated aging conditions. Oil-derived deposits on aftertreatment components are influenced not only by the total oil consumption, but also by the relative volatility of various oil components such as detergents and antiwear agents. Components in fresh oils may volatize faster than in aged oils, so replicating the number of oil changes is considered important. The application oil sump temperature also affects the rate of component volatilization, and should therefore be replicated as closely as possible during bench engine aging.

Processing Field Data Using the DAAAC Protocol

This section provides guidance on how to process the collected field data to derive an engine bench accelerated aging cycle. Where helpful, a long-haul truck application equipped with a V/W/TiO_2 Selective Catalytic Reduction (V-SCR) system and city bus application equipped with a V-SCR system are used as examples.

Engine Speed / Load

First, the modal engine speed data are normalized over the application engine speed range (idle to maximum engine speed, zero and 100 %, respectively) to provide Speed percent data. Second, the data are sorted into 5 % increment bins of speed and load range. Bins that represent less than 0.5 percent of the total operating time are then discarded. Figures 2 through 6 show the results of performing this operation on five sets of data representative of different types of applications. Per statistician recommendation, the data used must represent at least 80 percent of the field data in each case. The size of the bubbles represents the percentage of time spent within a given speed and load range. A cursory look at the data is all that is needed to see that none of these applications have similar operating duty cycles. Therefore, it is clear that different applications will frequently require different accelerated aging cycles.

Using statistical cluster analysis of the field data, aging modes are created to closely represent the field operation. The maximum number of modes in an aging cycle is set at 15. Using a long-haul truck V-SCR system as an example, field data were processed in this manner, resulting in a 15-mode aging cycle with the modes and times represented in Figure 7. The mode lengths were then normalized such that the cycle was exactly one hour in length.

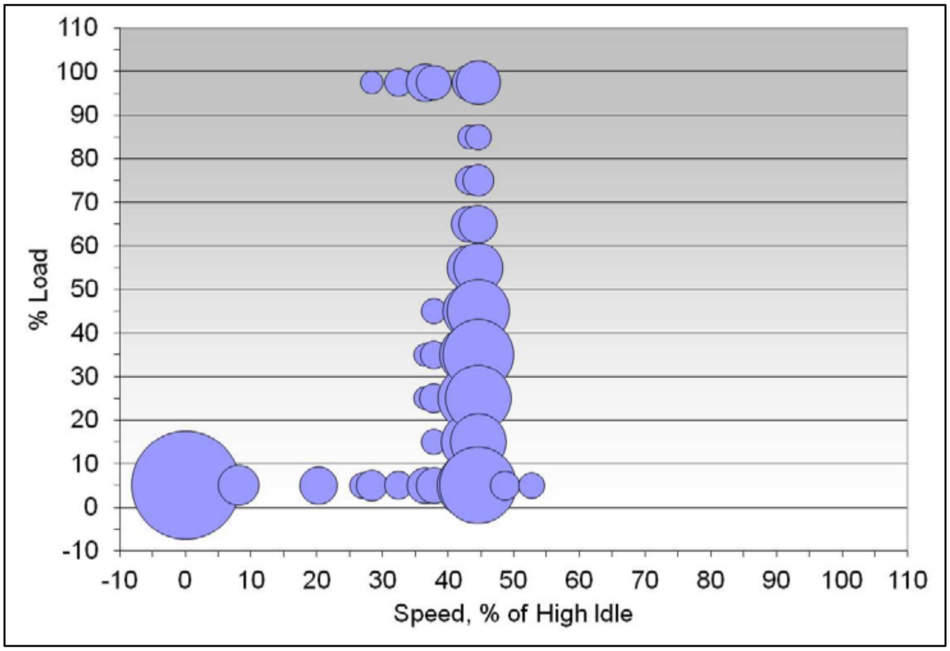

Fig. 2: Long-Haul Truck Application

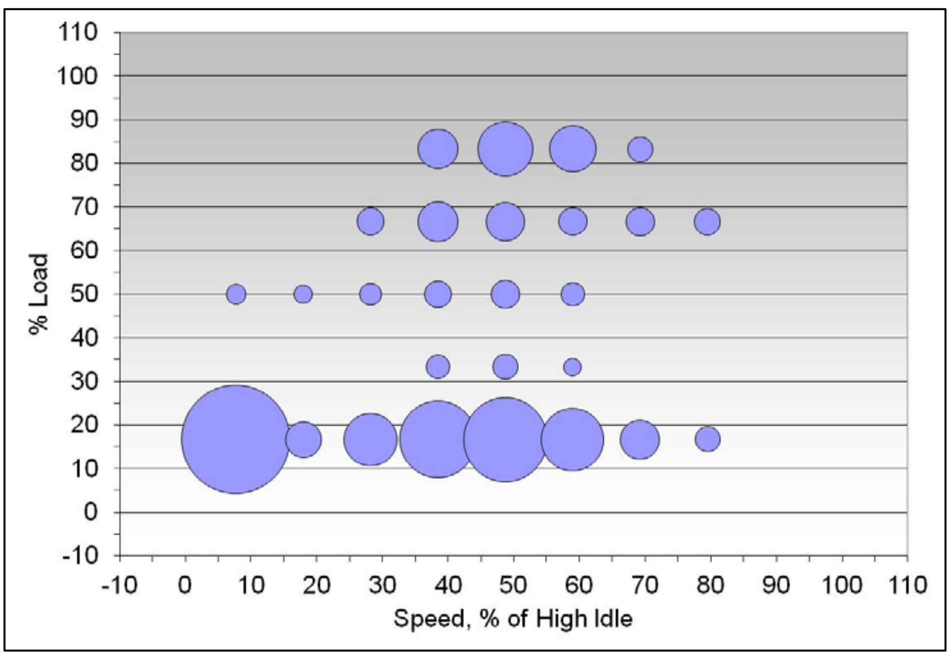

Fig. 3: City Bus Application

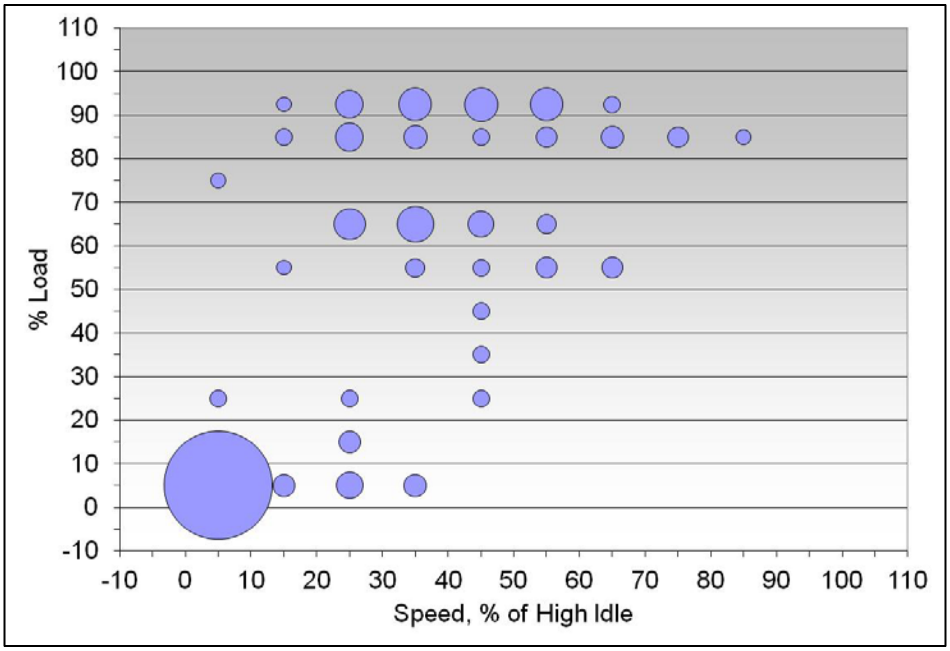

Fig. 4: Backhoe Loader Application

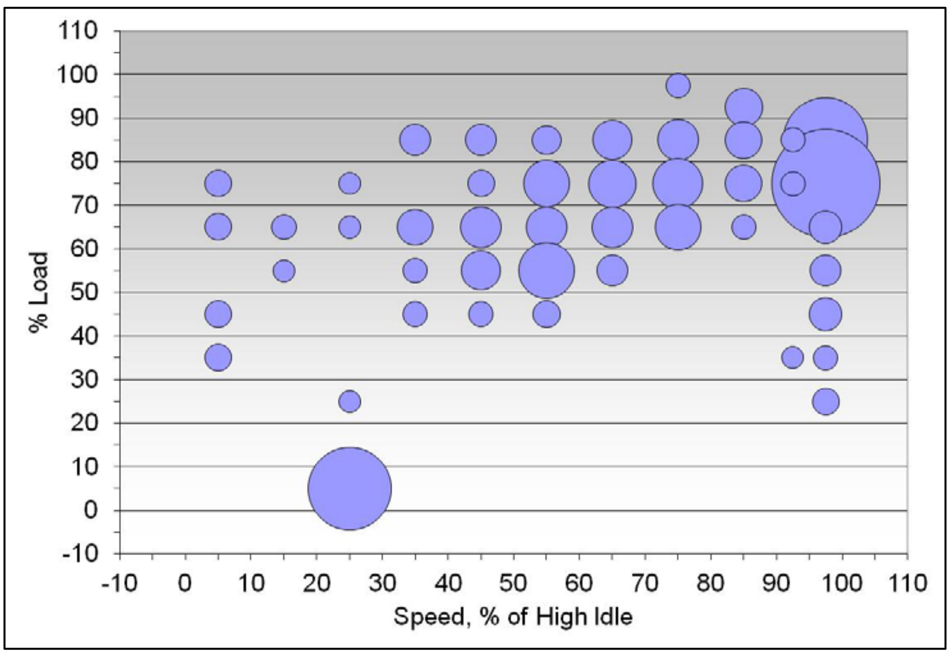

Fig. 5: Crawler Tractor Application

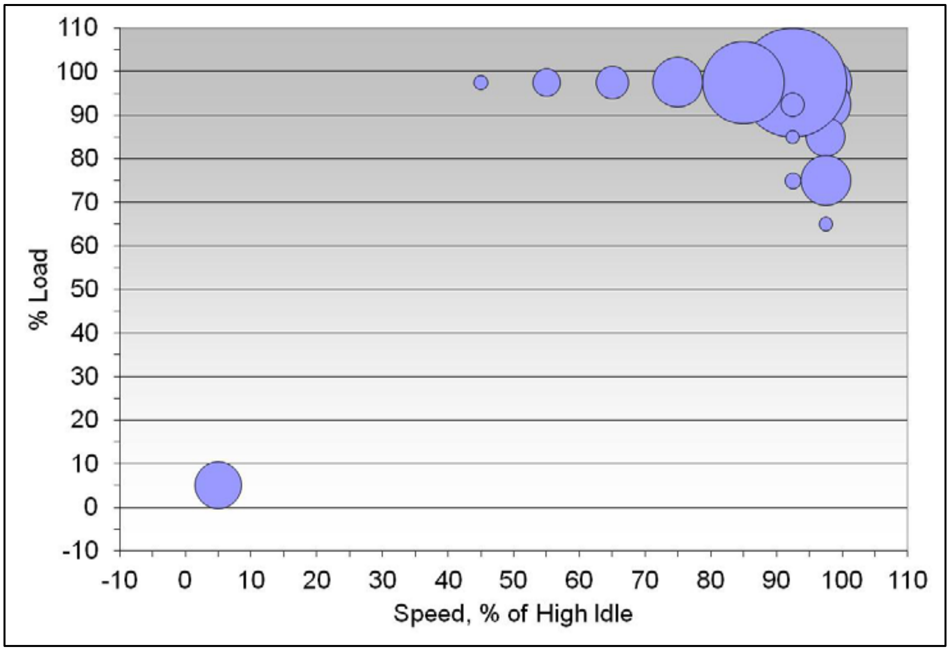

Fig. 6: Tractor Application

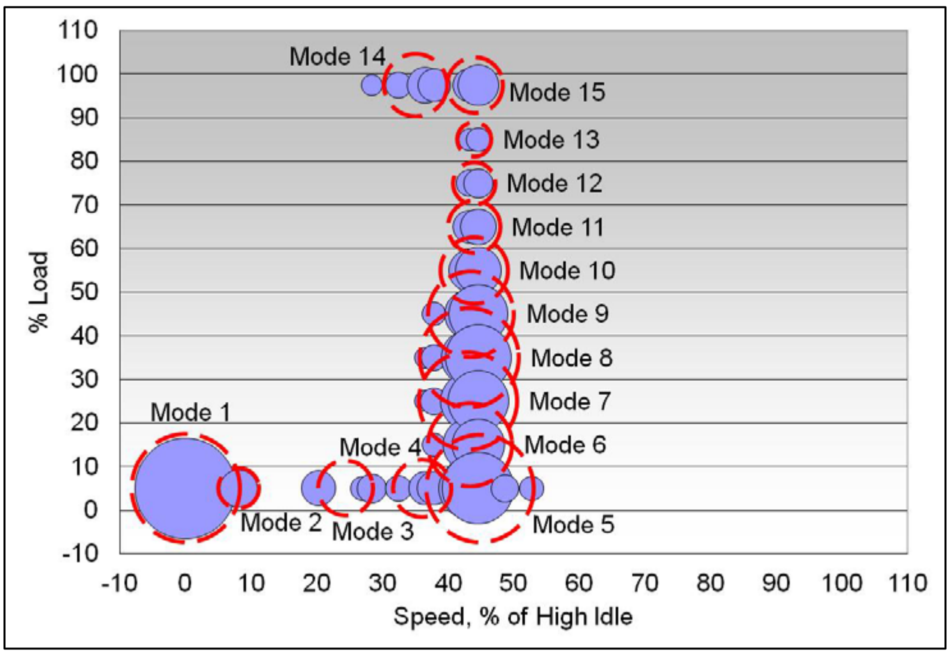

Fig. 7: Example 15-Mode Aging Cycle for Long-Haul Truck V-SCR System

Temperature Processing

Field temperature data are processed into bins of 10°C or smaller. The time in each bin is calculated, and then extrapolated out to the full useful life for the specific application. Some processed long-haul truck V-SCR system data are given as an example in Figure 8. In this case the data were only provided in 50°C bins, which would not be adequate. The total mileage of 617,864 km was calculated to represent 8,493 hours of operation.

LONG-HAUL TRUCK V-SCR SYSTEM FIELD DATA PROCESSING (SCR-S)												
DATASET 1	Data Mileage		51994 km		Total Mileage		617864 km					
Avg. SCR Inlet Temp	463	488	538	588	638	688	738	788	838	888		
Time @ Temp, Hrs	119	166	229	122	38	10	0	0	0	0	Total =	683
Total Time @ Temp, Hrs	1410	1971	2717	1453	449	115	4	0	0	0	Total =	8120
% of Operating Time	17	24	33	18	6	1	0	0	0	0		
DATASET 2	Data Mileage		31202 km									
Avg. SCR Inlet Temp	463	488	538	588	638	688	738	788	838	888		
Time @ Temp, Hrs	86	75	142	87	27	7	0	0	0	0	Total =	425
Total Time @ Temp, Hrs	1707	1481	2811	1730	543	134	7	0	0	0	Total =	8412
% of Operating Time	20	18	33	21	6	2	0	0	0	0		
DATASET 3	Data Mileage		17304 km									
Avg. SCR Inlet Temp	463	488	538	588	638	688	738	788	838	888		
Time @ Temp, Hrs	40	63	67	53	21	6	1	0	0	0	Total =	251
Total Time @ Temp, Hrs	1442	2235	2389	1887	758	213	25	0	0	0	Total =	8948
% of Operating Time	16	25	27	21	8	2	0	0	0	0		
AVERAGED DATA FROM THE THREE DATASETS												
Avg. SCR Inlet Temp	**463**	**488**	**538**	**588**	**638**	**688**	**738**	**788**	**838**	**888**	**per client - 10K**	
Total Time @ Temp, Hrs	**1520**	**1896**	**2639**	**1690**	**583**	**154**	**12**	**0**	**0**	**0**	**Total =**	8493
% of Operating Time	18	22	31	20	7	2	0	0	0	0		

Fig. 8: Example of Field Temperature Data Extrapolation

The data are then reduced down to a single temperature, and the effective aging time at that temperature is determined using Equation (2), which is based on the Arrhenius equation,

$$\frac{t1}{t2} = exp\left[-\frac{Ed}{R}\left(\frac{1}{T2} - \frac{1}{T1}\right)\right] \tag{2}$$

where *Ed* is the deactivation energy of the primary catalytic component in kJ/mol, *R* is the gas constant, t_1 is a known time, T_1 is the temperature (in degrees Kelvin) during time t_1, T_2 is the desired single temperature, and t_2 is the calculated equivalent time at T_2.

Figure 9 shows the example long-haul truck V-SCR system data reduced to a single temperature. In this example, a single point aging temperature (T_2) of 688K (415°C) was chosen. The temperature was selected in this case as being below the maximum recommended operating temperature of 500°C for V-SCR catalysts, and high enough to achieve aging in a reasonable time period. A deactivation energy (*Ed*) value of 150 kJ/mol was used. Suitable values of *Ed* for each catalyst type should be determined to provide the most accurate time at temperature correlations. The DAAAC Protocol does not define how these *Ed* values must be derived, leaving that to the discretion of the user. Bartley [2] presents a method for determining *Ed*, and provides experimentally derived values for various SCR formulations. In the example, it was determined that 323 hours at 688K would provide the same effective thermal aging as 8,493 hours under normal operating conditions.

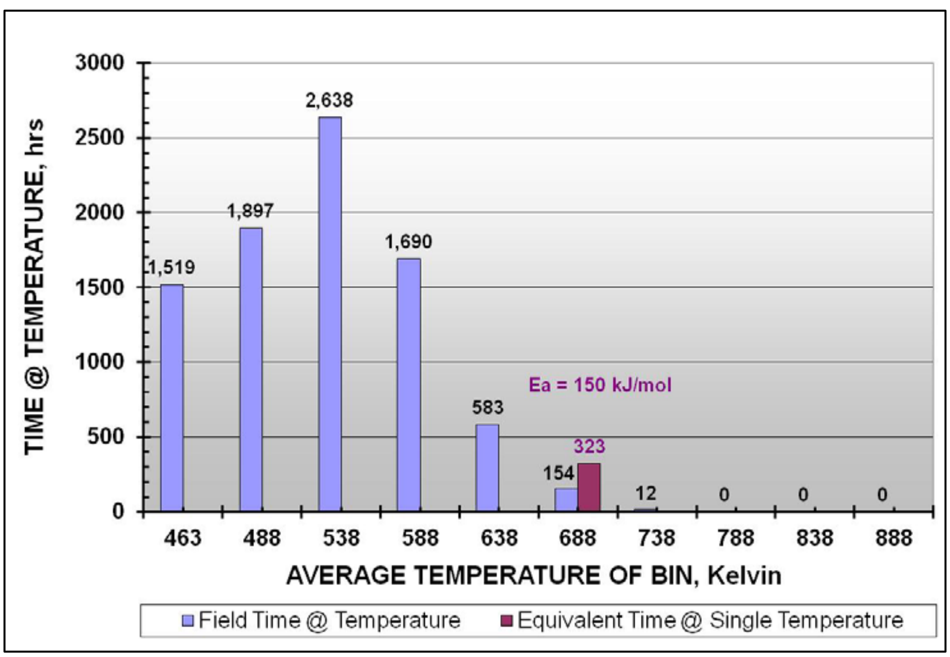

Fig. 9: Long-Haul Truck V-SCR System, Time at Temperature Correlation

The aging engine is then operated under the aging conditions defined for the application to measure the catalyst inlet/bed temperatures. Temperatures are measured at suitable locations (both inlet/outlet gas and internal catalyst bed temperatures). These data are then processed to determine the effective aging time relative to the selected single point temperature (688K for the V-SCR example). The total aging time required to match the single point aging time is then extrapolated. Figure 10 shows data from the example 15-mode cycle. Here, 8,493 hours of normal operation thermal aging can be recreated with 323 hours at the selected single aging temperature of 688K, or 177 hours over the 15-mode aging cycle temperature range. The entire process is illustrated in Figure 11.

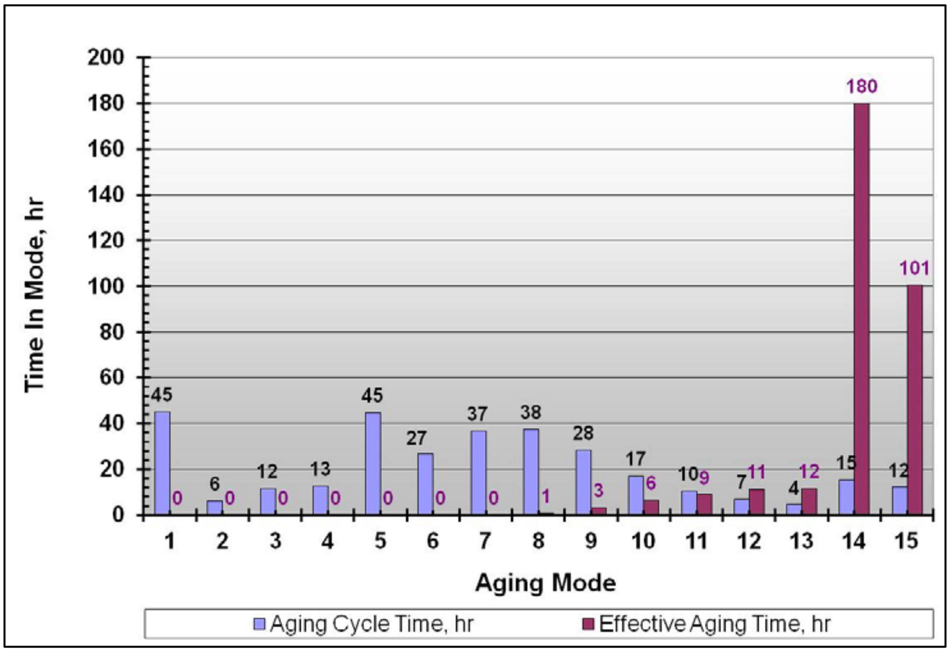

Fig. 10: Long-Haul Truck V-SCR System, Actual and Effective Aging Time at Each Mode

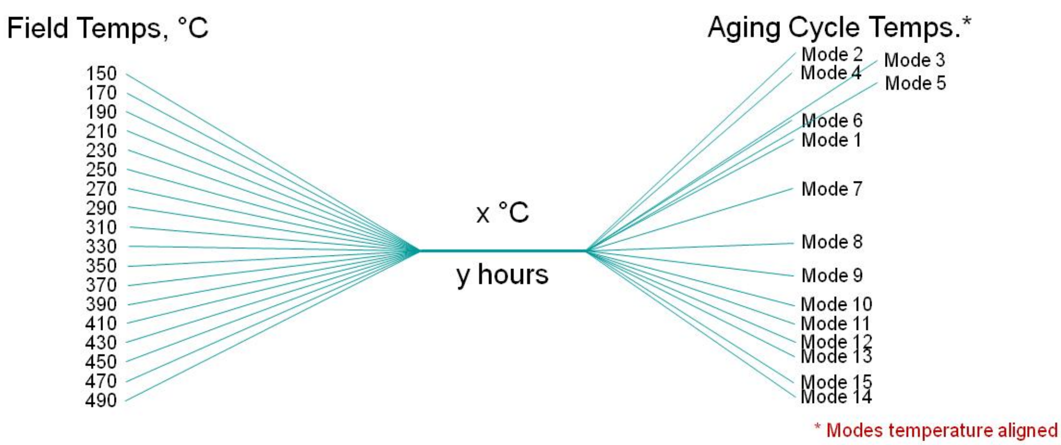

Fig. 11: Illustration of Field Temperature Reduction and Aging Temperature Expansion

Actual aging temperatures and times are strongly dependent on the bench aging configuration, and must be determined for each configuration and application.

Mode Sequencing

There is no required sequence for the aging modes. The modes may be sequenced by the user to reflect the application operation if so desired, but this has not been demonstrated to have any measurable effect on the final aging product. Experience with mode sequencing suggests that the simplest effective mode sequence is to order the modes from lowest to highest load, referred to here as a stepped load sequence. The stepped load sequence generally results in the highest overall aging cycle temperatures, which in turn results in the shortest total required aging time. Figure 12 provides an example of a stepped load sequence using the 15-mode V-SCR case.

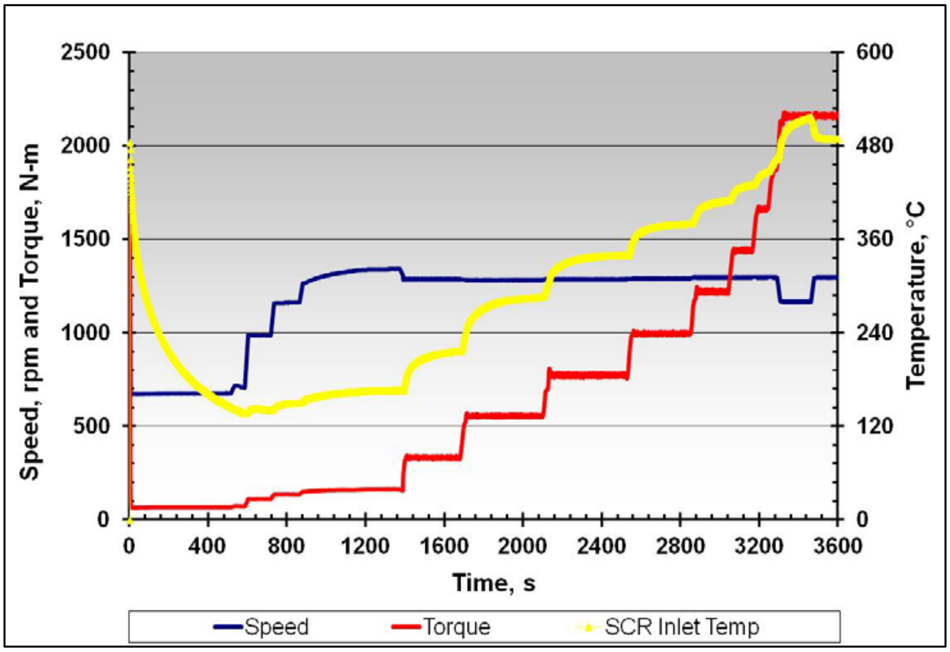

Fig. 12: Stepped Load 15-Mode Cycle Example

Control Temperature Considerations

Because the Arrhenius equation is an exponential equation, aging time is strongly dependent on aging temperature. As a consequence, even relatively small changes in aging temperature can have a significant impact on the effective aging time. For this reason, a software algorithm is used to calculate the cumulative effective aging time in real time. The Real Time-Bench Aging Time™ (RT-BAT™) algorithm, as described in [3] calculates the effective aging time for each temperature measurement, at a frequency of 1 Hz. For example, if the desired aging time is 177 hours, aging will be performed until the **effective** aging time is 177 hours, even if the actual aging time is more or less. Adopting this approach improves the precision of the aging by compensating for anything that may change the actual aging temperatures from those intended.

Temperature Control Thermocouple Location

Accelerated bench engine aging cycles for gasoline applications with three-way catalysts have been in existence for some time. The cycles often specify a desired aging temperature, and a thermocouple location where this control temperature is measured. Some cycles place the thermocouple upstream of the catalyst, thereby only measuring the exhaust gas inlet temperature to the catalyst. Others place the thermocouple at one location in the bed of the catalyst, thereby including catalyst exotherm characteristics as part of the aging. The latter makes sense because the bed of the catalyst may be significantly hotter than the inlet gas, and catalyst deactivation is directly correlated to the temperature the catalyst experiences. For illustrative purposes, an SCR catalyst was instrumented with one inlet and three bed temperature thermocouples, as shown in Figure 13.

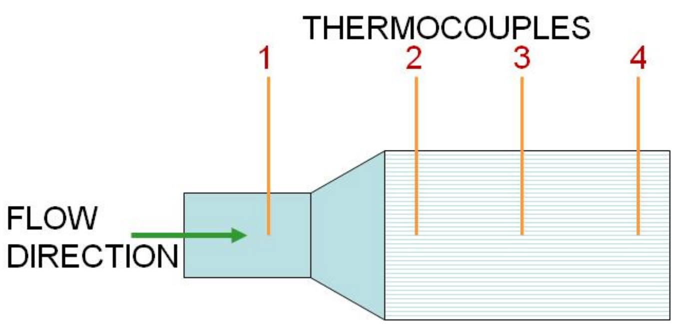

Fig. 13: Example Thermocouple Locations in SCR Catalyst

The example 15-mode aging cycle was run and all four temperatures recorded. Total aging times required to match the single point aging time were calculated from these data. For thermocouple locations 1, 2, 3 and 4, the calculated aging times were 177, 179, 209 and 174 hours. The aging times were reasonably similar for this SCR catalyst that had only a minor exotherm of about 5°C. Even so, the mid-bed location 3 had lower average temperature over the full 15-mode cycle. These data demonstrate the importance of control thermocouple location selection. Differences are much larger for aftertreatment systems with larger exotherms, such as DOCs and DPFs.

If bed temperature is used, a logical option is to place the thermocouple at the highest temperature location within the catalyst because this location experiences the most aging. However, a complicating factor with catalyst bed temperatures is that the peak temperature location will be in different locations for different catalyst formulations and exhaust conditions. One option is to require that the thermocouple be placed at the highest bed temperature location, to be determined by measuring bed temperatures at various locations within the catalyst while generating the field data. This would need to be done for every system configuration and catalyst type. The RT-BAT calculator is useful in this regard. If multiple thermocouples are located within the catalyst bed, only the highest temperature need be used by the RT-BAT calculator to add to the incremental effective aging time total. There is another advantage to this approach. The location of the peak bed temperature may migrate within the catalyst as the catalyst ages. This is illustrated in Figure 14 by the bed temperature profiles within a DOC after different periods of aging. Using the RT-BAT calculator accommodates this aging effect by always calculating effective aging time using the highest temperature only. This approach is called Multipoint Temperature Monitoring™ (MTM™), and is described in [3].

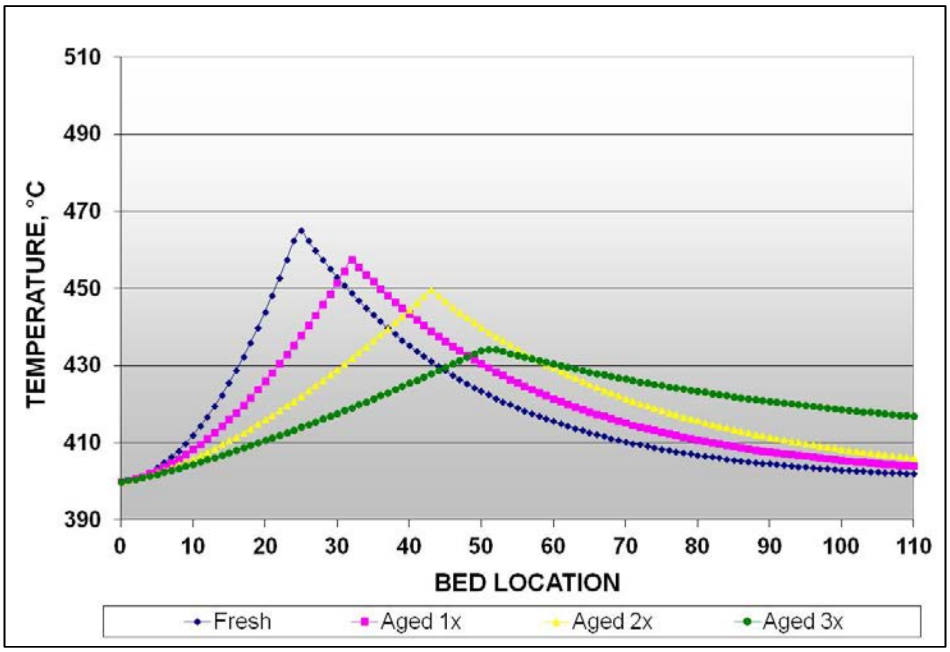

Fig. 14: Illustration of Bed Temperature Profile as a Function of Aging

Another problem with single point bed temperature control is less obvious, but very significant to catalyst aging. Single point bed temperature control requires that the bed temperature at one location be maintained at the required temperature throughout aging. If the exotherm at that location decreases with aging time due to catalyst deactivation, forcing the temperature back up to the required value may lead to even higher, unrealistic temperatures elsewhere in the catalyst. This is illustrated in Figure 15. Use of RT-BAT with MTM eliminates a number of problems, and is required in operation of the DAAAC Protocol.

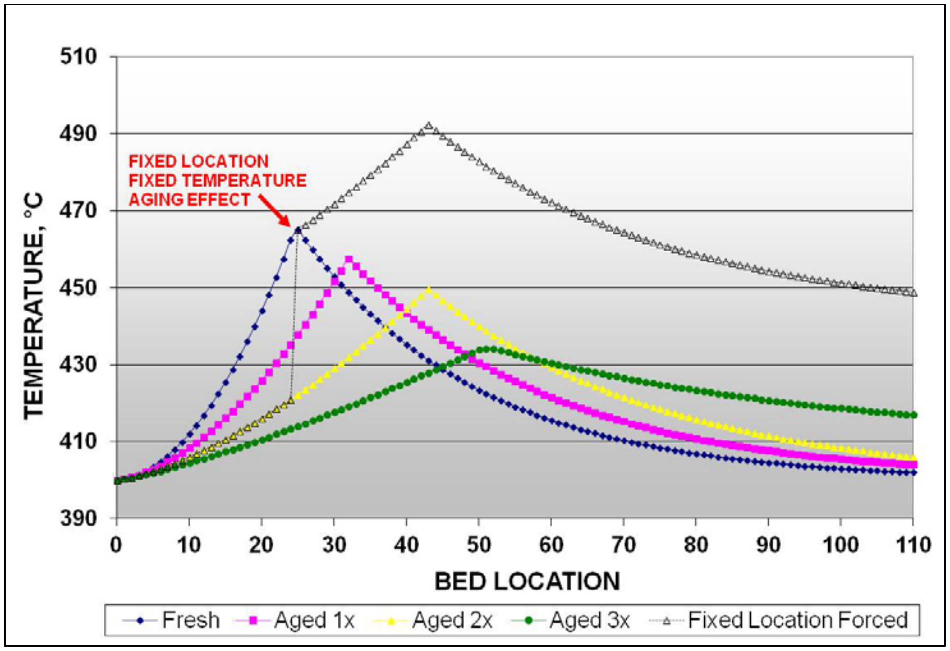

Fig. 15: Effect of Single Point Aging Control on Other Bed Temperatures [3]

Oil Consumption

A more difficult, but important aging mechanism is the effect of deposits on the aftertreatment system components. Inorganic and semi-metallic elements in lubricating oil are known to have an adverse effect on catalyst activity in some cases. The oil consumption information from the field data is used to provide input into the accelerated aging procedure. It is understood that the total aftertreatment component exposure to non-organic oil components is more important than just the volume of oil consumed. Therefore, the levels of phosphorus, zinc, calcium, magnesium, boron and sulfur in the field and aging oils, and their actual composition must be taken into account. The best way to do this is to use the same oil for the accelerated aging as was used in the field data collection. This is a requirement of the DAAAC Protocol. Ideally, the oil will all be from a single batch.

The total oil consumption and oil change intervals are recorded during the field operation. Oil top offs should be avoided during the field operation, but must be recorded and factored into the total oil consumption calculations if they do occur.

The oil consumption rate of the aging engine is measured under the aging cycle conditions. The number of aging cycles required to reach full useful life total oil consumption can then be calculated. It is highly likely that the bench engine oil consumption rate during the thermal aging cycle will not be sufficient to achieve the desired total oil consumption in the same time/cycles as needed to achieve the thermal aging target. There are various methods available to increase the oil consumption rate, or oil component consumption rate [4] [5] [6] [7] [8]. To further increase the oil consumption without affecting the thermal aging, the addition of an oil mode is permitted. A typical oil mode operates at a condition that promotes oil consumption, but has significantly lower component temperatures such that the total effective thermal aging increases only slowly during the oil mode. Addition of an oil mode will lengthen the total aging time required, but may well be necessary to achieve both the thermal aging and oil consumption targets at the same time.

Correlation of Field- and DAAAC-Aged Aftertreatment Systems

Emissions tests were conducted on degreened (as a baseline), field aged and bench engine aged systems (using the DAAAC Protocol) to compare their performance. If the bench engine aging is successful, the emissions performance of the bench aged systems will match that of the field aged systems. Emissions performance tests and sound physical analyses were used to correlate field-aged and bench-aged V-SCR systems from a city bus application. Full bench-engine emissions tests were performed on a degreened (used as a baseline), field-aged and bench-aged system. Subsequently, various physical analyses were performed on the catalysts to compare the characteristics of the field- and bench-aged catalysts.

Emissions test cycles included the HD-FTP (cold and hot), WHTC (cold and hot), RMC, ESC and WHSC. The degreened city bus SCR was used to determine DEF injection for all test cycles. The DEF injection strategy utilized for the performance evaluations maintained tailpipe NH_3 slip at or below 15 ppm for the degreened system. Hence, the ammonia-NOx ratio (ANR) was frequently less than 1 and the catalyst conversion efficiencies were quite low. Prior to conducting the series of performance tests, each

SCR catalyst was conditioned on the emissions test cell engine at 400°C with no DEF injection for four hours. Conditioning was used to remove any stored NH_3 from the catalyst. The results are shown in Figure 16.

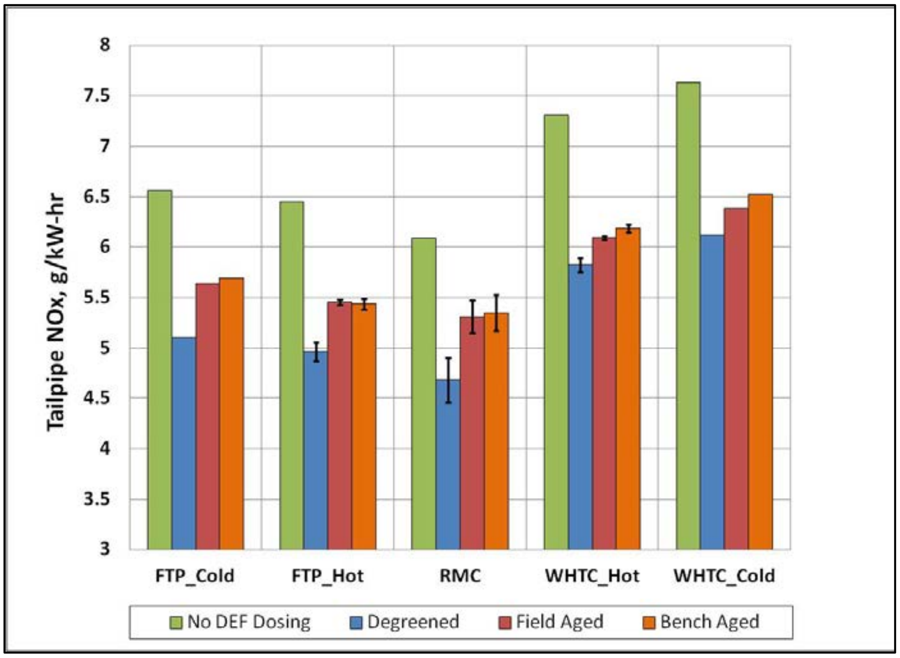

Fig. 16: City Bus Application V-SCR Performance Results

Where multiple tests were performed, error bars are included to show the test repeatability and indicate significant differences. For the FTP_Cold, FTP_Hot and RMC tests, the field and bench-aged catalysts performed similarly, with significantly lower NO_x conversion efficiencies than the degreened catalyst. The WHTC Cold and Hot tests were similar, with less differentiation.

BET surface areas were obtained from catalyst samples of each city bus V-SCR system. Results are shown in Figure 17. Despite individual sample surface area variations, possibly from uneven washcoating, sampling or measurements, the average results were statistically equivalent, indicating no significant thermal aging.

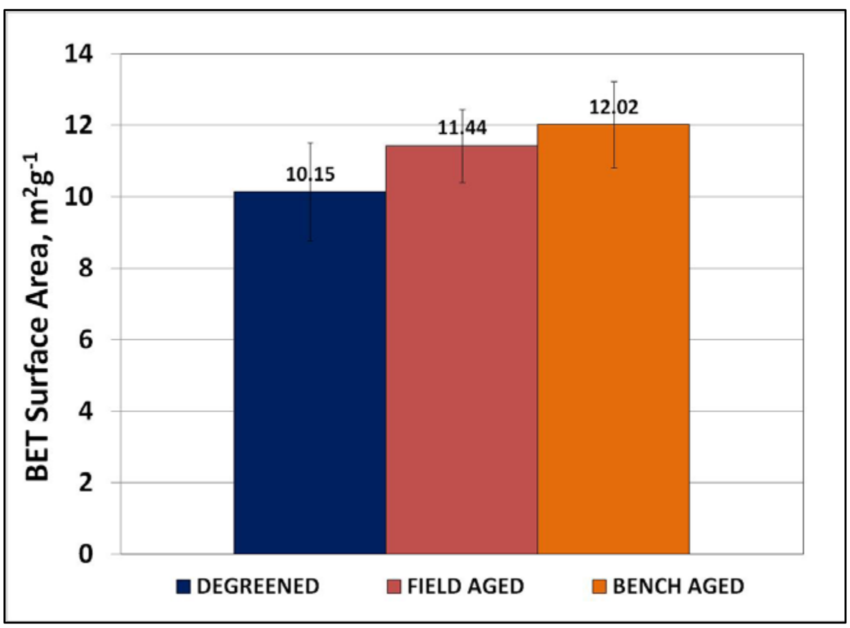

Fig. 17: BET Surface Area Measurements

Proton Induced X-Ray Emission (PIXE) analyses were conducted to compare oil deposit profiles in the degreened, field aged, and bench aged samples. As can be seen in Figure 18, there was a clear phosphorous deposition profile with decreasing levels from the inlet of the front catalyst to the outlet of the back catalyst, where the level was below detection limits. The highest P level found on the inlet of the front catalyst along the centerline was about 0.47 wt%. From the PIXE analysis, it may be deduced that the field-aged system was exposed to more oil consumption than indicated by the fleet average field data, however, the deposition profiles between the field aged and bench aged systems were similar. The city bus system supplier provided information that the fleet average oil consumption value was based on a fleet of more than 100 vehicles (variation in fleet oil consumption was not provided).

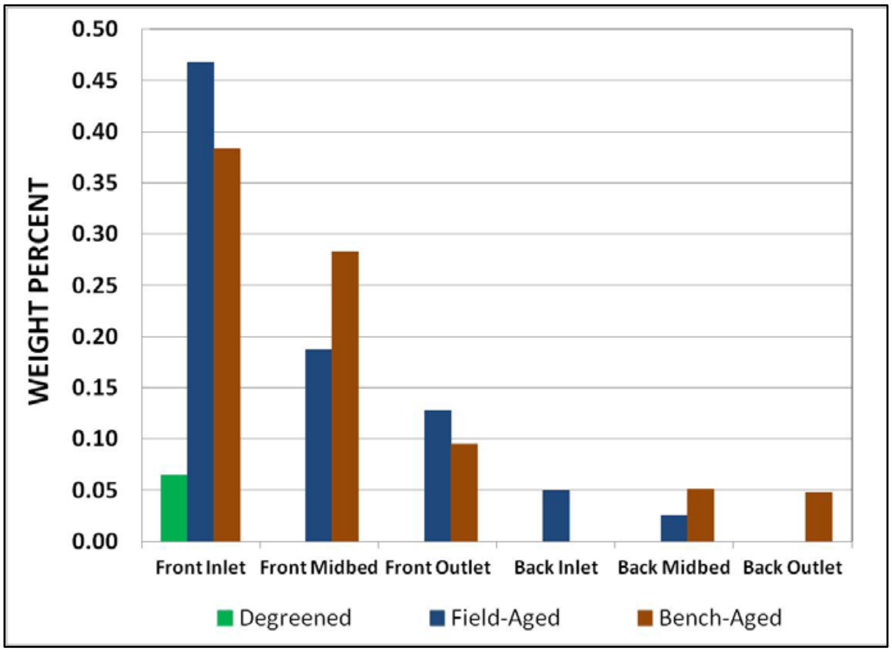

Fig. 18: PIXE Analysis Results for Field-Aged and Bench Aged City Bus V-SCR Systems – Phosphorous Oil Component

Scanning Electron Microscopy (SEM) and Energy Dispersive Spectrometry (EDS) were performed on equivalent samples to those subjected to the PIXE analysis. Images were obtained at magnifications of between 1000x and 6000x, concentrating on the phosphorus deposits, and are shown in Figure 19. It was apparent the larger oil deposit particles only sparsely populated the surface. In the images presented, an oval in the image indicates the particles of oil deposits (phosphorus, calcium, sulfur). Sulfur is strongly associated with the oil deposits, which is very reasonable since sulfates are common compounds identified in oil ash deposits. When found, the deposits appear to have a spherical morphology for both the field- and bench-aged samples.

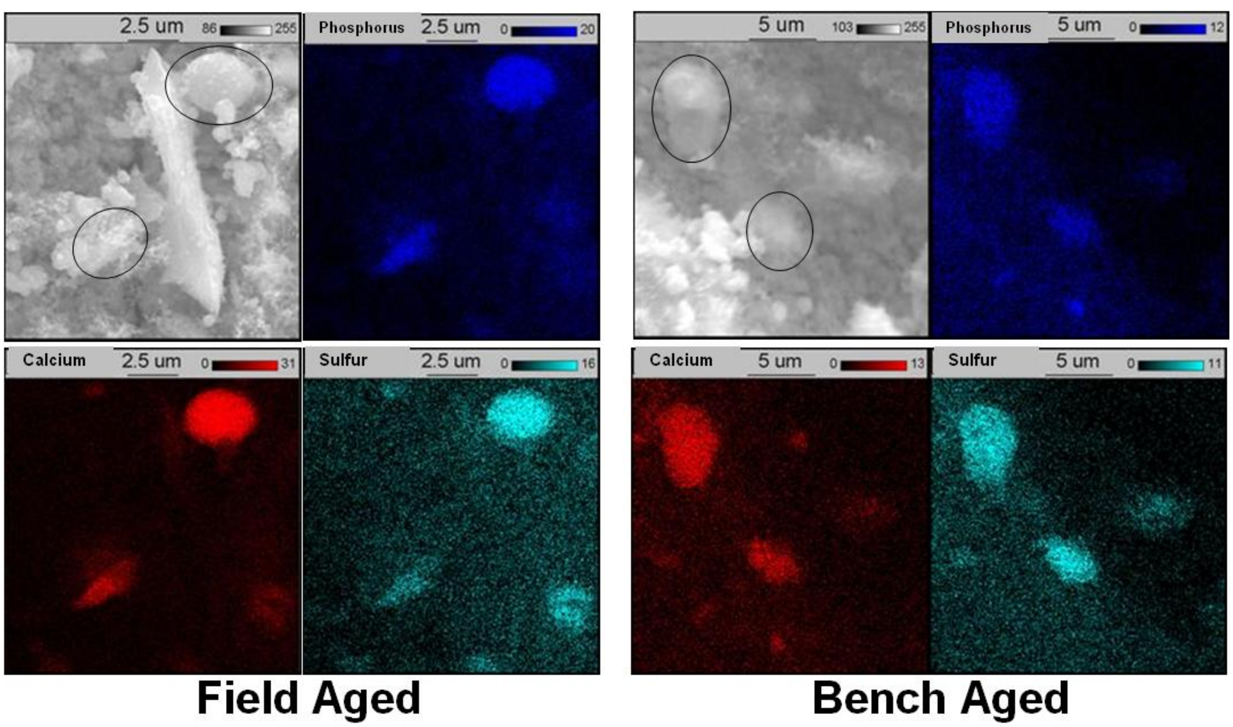

Fig. 19: SEM / EDS Images for Field-Aged and Bench-Aged City Bus V-SCR Systems

Conclusion

The DAAAC Protocol is an accelerated aging procedure for diesel aftertreatment ***systems***. This procedure has demonstrated aftertreatment system deterioration similar to that experienced in the field. The Protocol can be useful for aftertreatment system development programs and will help in determining deterioration factors. The procedure could also be employed in relevant future regulations for accelerated aging of diesel aftertreatment systems.

This paper presented the procedures used behind the Protocol: field data collection, field data processing, preliminary DAAAC aging cycle definition, bench engine setup, preliminary aging cycle operation with data collection, bench engine data processing, and final DAAAC aging cycle definition. Emissions performance tests and physical analyses were used to correlate field-aged and bench-aged (using the DAAAC Protocol) aftertreatment systems. The combined data demonstrated good correlation between the field- and bench-aged catalysts.

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