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NOx Adsorber Performance in a Light-Duty Diesel Vehicle

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ABSTRACT

Light-duty chassis dynamometer driving cycle tests were conducted on a Mercedes A170 diesel vehicle with various sulfur-level fuels and exhaust emission control systems. Triplicate runs of a modified light-duty federal test procedure (FTP), US06 cycle, and SCO3 cycle were conducted with each exhaust configuration and fuel. The fuels used in these experiments met the specifications of the fuels from the DECSE (Diesel Emission Control Sulfur Effects) program (1-4)[1]. Ultra-low sulfur (3 ppm) diesel fuel was doped to 30 and 150 ppm sulfur so that all fuel properties except sulfur content would be the same.

Although the Mercedes A170 vehicle is not certified for sale in the United States, its particulate matter (PM) and nitrogen oxide (NO_x) emissions in the as-tested condition were within the Environmental Protection Agency's Tier 1 full useful life standards with its OEM oxidation catalysts installed. Engine-out tests showed that the OEM catalysts reduce PM by 30-40%. There was very little effect of fuel sulfur on the performance of the OEM oxidation catalysts.

With a replacement lightoff catalyst and NO_x adsorber (lean-NOx trap) installed, FTP NO_x emissions were reduced by more than 90% with 3 ppm fuel. Reductions of NO_x on the US06 and SC03 cycles were 89% and 96%, respectively. Following the evaluations with the ultra-low sulfur fuel, the catalyst system was poisoned with the equivalent of 3,000 miles on 30 ppm sulfur fuel, which reduced the system's effectiveness to 80% on the FTP, 66% on the US06, and 84% on the SC03.

INTRODUCTION

The pursuit of increasing vehicle fuel economy has focused attention on lean burn engines in recent years. The diesel engine has been challenged to achieve NO_x and PM emissions similar to gasoline engines. One enabling technology for the diesel and other lean-burn engines is the NO_x adsorber. The NO_x adsorber has the potential to greatly lower tailpipe NO_x emissions from lean-burn engines, but it has also been shown to be very sulfur sensitive (1).

The Diesel Emission Control Sulfur Effects (DECSE) program evaluated the performance of several diesel emission control technologies with ultra-low sulfur No. 2 diesel fuel doped to various sulfur levels. All performance evaluations were conducted on stationary dynamometer test stands, and all NO_x adsorber tests were at steady state conditions. Tests showed the effects of fuel sulfur on device performance and particulate emissions. Devices evaluated under the auspices of DECSE included diesel oxidation catalysts, lean NO_x catalysts, continuously regenerating diesel particulate filters, catalyzed diesel particulate filters, and NO_x adsorbers (1-4). The vehicle tests discussed in this paper were intended to enhance the DECSE program by evaluating NO_x adsorbers during *transient* light-duty driving cycles. The goals were to quantify the emissions reduction potential of some of these near-term emission control devices and to examine the effects of fuel sulfur on their performance. A companion paper discusses a catalyzed diesel particulate filter evaluation with this same vehicle (5).

The Diesel Vehicle Emission Control Sulfur Effects (DVECSE) project was designed to augment and build on the DECSE program. Time and resource limitations constrained the first phase of DVECSE to one NO_x adsorber and one diesel particulate filter evaluation. The vehicle's baseline emissions were measured for both the engine-out condition and with original equipment manufacturer (OEM) catalysts installed.

The three-way catalyst has enabled monumental reductions in carbon monoxide (CO), hydrocarbons (HC), and NO_x from stoichiometric engines. However, because of the excess oxygen in lean-burn engine exhaust, the conventional three-way catalyst is ineffective at reducing NO_x. Many researchers have reported on the promise of the NO_x adsorber to control NO_x emissions from lean-burn engines (1,8-28). Most of these have focused on gasoline engines (8-26), but a few have investigated its use in diesels (1,27,28). The low temperature exhaust of light-duty diesels provides a challenge to the effective use of the NO_x adsorber with this powerplant.

[1] Numbers in parentheses refer to references at the end of the paper

Additionally, generating the fuel-rich exhaust conditions required for regeneration can be more difficult than with lean-burn gasoline engines.

RESEARCH VEHICLE

The testbed for the reported work was a Mercedes A170 CDI, shown in Figure 1. The vehicle's curb weight was 1,095 kg, as equipped with a 5-speed manual transaxle and 1.7 liter diesel engine. The engine is a turbocharged, intercooled, 4-valve-per-cylinder, direct-injection, common-rail design that produces 66 kW at 4,200 RPM. The engine utilizes exhaust gas recirculation (EGR) for NO_x control, plus two oxidation catalysts to control CO, HC, and PM emissions. The first catalyst is close-coupled directly to the outlet of the turbocharger, with the second catalyst mounted in an underfloor configuration.

Figure 1. Mercedes A170 research vehicle

After its delivery from Europe, the vehicle was sent to a nearby research facility for break-in mileage accumulation (6,500 km) on a closed-course under prescribed conditions. The break-in mileage accumulation was conducted using ultra-low sulfur fuel. Following the break-in mileage, the vehicle was instrumented at ORNL for relevant temperatures, pressures, accelerator pedal position, engine speed, and other measurements, which were collected using an in-vehicle data acquisition system. This same data acquisition system was also used to monitor laboratory instruments during all chassis dynamometer tests.

TEST FUELS

Phillips ultra-low sulfur diesel fuel was used for engine and catalyst break-in, and the same fuel doped to various sulfur levels was used in all evaluations. The fuel specifications were the same as those from the DECSE program. Some relevant fuel properties are show in Table 1; a thorough discussion of the DECSE fuels can be found in reference 2. The OEM catalysts and engine-out evaluations were conducted with 3, 30, and 150 ppm sulfur fuels. The NO_x adsorber was evaluated with 3 and 30 ppm fuels. The 30 ppm NO_x adsorber tests were preceded by an accelerated sulfur loading with 150 ppm fuel. The catalyzed diesel particulate filter tests discussed in reference 5 were also conducted with 3, 30, and 150 ppm sulfur fuels.

VEHICLE DYNAMOMETER EVALUATIONS

All driving cycle emissions tests were conducted in triplicate on a twin-roll eddy current chassis dynamometer, which has been shown to effectively simulate FTP and US06 certification tests (6). A full-flow constant volume sampler dilution tunnel was used for collecting particulate and bag emissions. Emissions benches with the standard heated chemiluminescence NO_x analyzers, heated flame ionization HC detectors, paramagnetic oxygen detectors, and non-dispersive infrared CO and CO_2 analyzers were used to monitor raw and dilute exhaust gas during all evaluations. Additionally, a pulsed ultraviolet sulfur dioxide (SO_2) instrument was used for some of the tests.

A modified federal test procedure (FTP) was used to enhance particulate sample collection. The FTP calls for 3 phases, or bags. Bag 1 is the first 505 seconds, which includes a cold start; bag 2 is the next 867 seconds. The vehicle is soaked for 10 minutes, and the bag 1 drive cycle is repeated for bag 3. The calculation of the composite emissions from the 3 bags assumes that a 4th bag identical to bag 2 is collected (7). The modified FTP consists of only 2 phases, the first being a composite of bag 1 and bag 2, and the 2nd being a composite of bag 3 and bag 4. Given that each phase of the modified FTP is 1,372 seconds long, two larger particulate samples can be collected rather than three smaller ones. This 1,372 second cycle is also referred to as the LA4, and is shown in Figure 2. Triplicate runs of the modified FTP were conducted for each fuel and exhaust configuration.

The high-speed, high-load US06 cycle was conducted immediately following the second phase of the modified FTP. The 600 second US06 cycle is shown in Figure 3. The SC03 "air conditioning" cycle was run following an FTP bag 1 ("hot 505") preconditioning. The SC03 cycle was run at ambient laboratory conditions (nominally 75°F). The test vehicle was not equipped with air conditioning, so engine loads were not representative of an actual SC03. However, because the SC03 has different transients than the FTP and US06, the vehicle was evaluated on this cycle nonetheless. The 598 second SC03 cycle is shown in Figure 4.

Table 1. Base Fuel Specifications (2)

Property	ASTM Test	As Measured
Density, kg/m^3	D1298/ D4052	826.1
Viscosity (40C) mm^2/s	D445	2.42
Distillation IBP, C	D86	185
5% recovery, C	D86	198
10% recovery, C	D86	207
20% recovery, C	D86	222
30% recovery, C	D86	238
40% recovery, C	D86	251
50% recovery, C	D86	259
60% recovery, C	D86	266
70% recovery, C	D86	274
80% recovery, C	D86	287
90% recovery, C	D86	314
95% recovery, C	D86	338
FBP	D86	350
Carbon, mass %	D5291	86.3
Hydrogen, mass %	D5291	13.4
Sulfur, ppm	D4045 D5453	3.1
Saturates, vol. %	D1319	70.7
Olefins, vol %	D1319	2.3
Aromatics, vol. %	D1319	27.0
Aromatics, wt. %	D5186	28.5
Polyaromatics, wt. %	D5186	9.6
Non-aromatics, wt. %	D5186	71.2
Heat Comb, net, MJ/kg	D240	43.1
Cetane number	D613	44.8
Cetane index	D976	53.6
Cloud point, C	D2500	-21.0
HFRR lubricity, µm (without lubricity additive)	D6079	635
HFRR lubricity, µm (with 205 ppm Octel FOA 35a lubricity additive)	D6079	355

NO_X ADSORBER REGENERATION

The NO_X adsorber is a flow-through emissions control device that can adsorb nitrogen oxides during lean operation. Before the unit is saturated with NO_X, a fuel-rich excursion is required to both release the NO_X and chemically reduce it by three-way catalysis. It is expected that future applications of this technology on diesels will utilize advanced engine controls to periodically regenerate the device. Late-cycle, in-cylinder injection of excess fuel can probably achieve the necessary rich condition and elevated temperatures necessary for regeneration (1). Because of the limited commercial availability of programmable diesel engine controls, and because of the extensive engineering required to achieve appropriate engine operation over transient drive cycles, the NO_X adsorber in the described work was regenerated with synthesis gas (syngas) injected into the exhaust system (Figure 5).

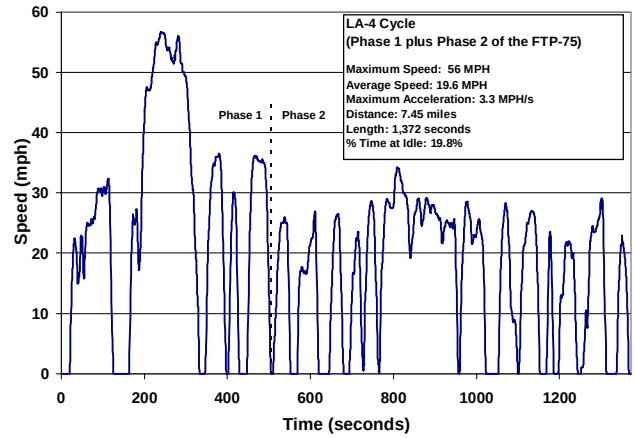

Figure 2. LA4 drive cycle

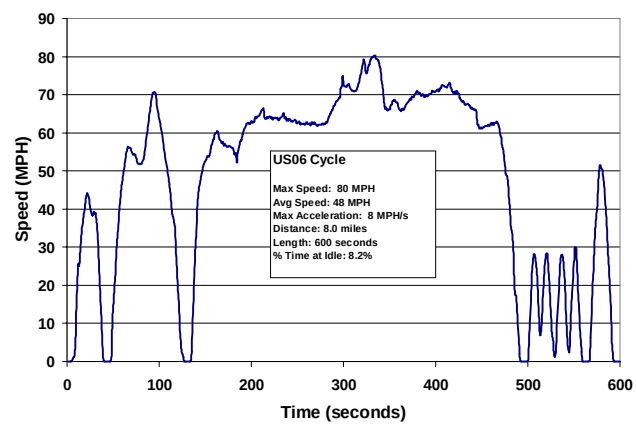

Figure 3. US06 drive cycle

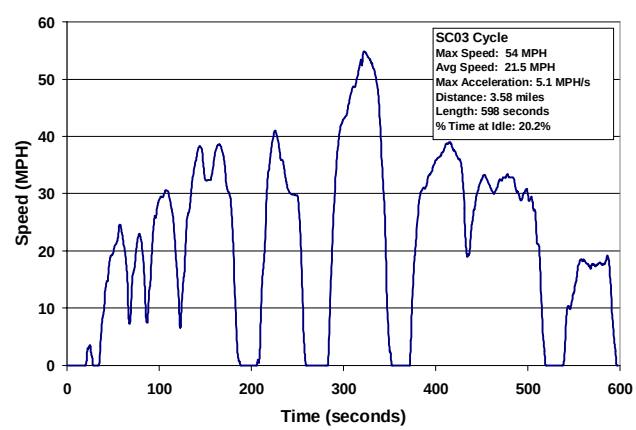

Figure 4. SC03 drive cycle

To simulate late-cycle, in-cylinder injection, the syngas was injected upstream of a lightoff catalyst. Syngas was composed nominally of (mole percent) 64-66% CO, 31-32% hydrogen (H_2), and 2-5% ethylene (C_2H_4). The lightoff catalyst oxidized the majority of the syngas to raise the temperature and significantly lower the oxygen

content of the exhaust entering the NO_x adsorber. To enhance the low temperature oxidation of the syngas, the stock Mercedes catalyst unit was removed from its housing and replaced with a new lightoff catalyst, provided by the manufacturer of the NO_x adsorber. The syngas was injected between the turbocharger exit and the close-coupled lightoff catalyst. Combustion of the H_2 was generally audible at injection. In tests with pure CO syngas no audible combustion was noted, and considerably lower temperature rises were recorded. The pure CO case will be discussed in more detail later.

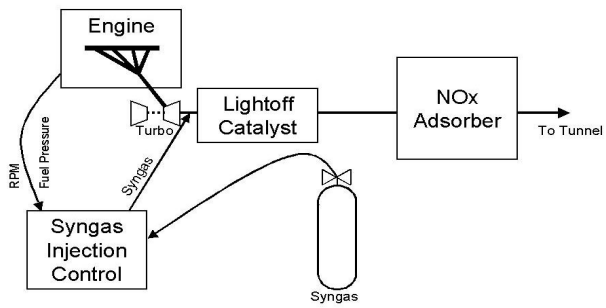

Figure 5. Syngas Injection System

The NO_x adsorber was physically located about 2 meters downstream of the lightoff catalyst in a typical underfloor location. A schematic of the exhaust system is shown in Figure 5. The NO_x adsorber was on a cylindrical 62 cell per square centimeter (400 cell per square inch) monolith, with a total volume of 2.5 liters. The 1.6 liter lightoff catalyst was in the factory housing on an oval monolith (also 62 cells per cm^2).

Synthesis gas demand was defined as the volume of synthesis gas necessary to establish a fuel-rich condition in the exhaust. The injected synthesis gas had to consume all excess exhaust gas oxygen leaving some CO available for NO_x desorption and reduction. The syngas injection control developed for this project utilized an aftermarket engine controller to meter the synthesis gas flow through four fuel injectors from a gaseous fuel application. The syngas control system monitored the engine speed (revolutions per minute, RPM) and fuel rail pressure in parallel with, but independent of, the Mercedes factory engine control unit (ECU). While the factory ECU controlled the engine independent of the syngas control, the syngas control periodically injected syngas into the exhaust independent of the engine control. Changes in engine load result in changes in RPM or fuel rail pressure, which subsequently cause changes in exhaust oxygen content, exhaust mass flow, and thereby syngas demand. The nonlinear nature of the syngas demand and the linear nature of the aftermarket engine controller made optimization of the system at all engine conditions difficult. The primary goals of the project were to demonstrate NO_x reduction on transient dynamometer drive cycles and to examine the effects of sulfur on the system performance. While minimizing fuel economy penalty or CO emissions are important for commercial viability of the technology, it was decided that these were secondary goals that would not be optimized within the scope of this phase of this project.

The syngas injection system was switched on for two seconds periodically to regenerate the adsorber at nominal 1 minute intervals (typically 50-90 seconds). The regeneration strategies were developed in a very short time frame, and are not considered to be optimal. With more development, it is expected that high NO_x conversion could be achieved with low CO and HC emissions and lower fuel economy penalties.

Figure 6 shows the combined LA4-US06 drive cycle and the injection strategy used in this program. Mid-bed temperature of the lightoff catalyst and NO_x adsorber were measured with 0.8 mm thermocouples inserted into one of the center flow channels of each monolith. These temperatures are also shown in Figure 6, as well as the CO and HC concentrations of the exhaust gas entering the adsorber, and the NO_x concentration entering and exiting the adsorber ("engine-out" and "tailpipe", respectively). The inconsistent CO and HC emissions with each regeneration event clearly indicate that a more refined control system could maintain NO_x control with more acceptable CO and HC emissions and fuel economy penalty.

SYNTHESIS GAS COMPOSITION – The original plan for regenerating the NOx adsorber was to employ in-pipe injection of diesel fuel, similar to the approach used in many diesel lean-NO_x catalyst experiments. After baseline tests of the vehicle revealed the particularly low exhaust temperatures, it was decided that synthesis gas would more accurately simulate in-cylinder injection for these experiments. The first approach was to specify a synthesis gas with the same H:C ratio as the diesel fuel being used (1.84:1). To achieve this H:C ratio, early development tests used a $CO:H_2$ ratio of 52:48. Excellent results could be obtained with this gas mix, although it was not possible to deliver enough gas under all conditions on the US06 to always insure a rich excursion. In an effort to decrease the explosive reactivity of the syngas and to decrease the large volumes of gas required, a blend of 56% CO, 19% H_2, and 25% C_2H_4 was tested next. The large ethylene fraction essentially cut the syngas demand in half because of the much higher stoichiometric air:fuel ratio, as shown in the following equations:

$$CO + \tfrac{1}{2} O_2 \rightarrow CO_2 \quad (1)$$

$$H_2 + \tfrac{1}{2} O_2 \rightarrow H_2O \quad (2)$$

$$\tfrac{1}{2} C_2H_4 + 1\tfrac{1}{2} O_2 \rightarrow CO_2 + H_2O \quad (3)$$

$$CH_{1.84} + 1.46\, O_2 \rightarrow CO_2 + 0.92\, H_2O \quad (4)$$

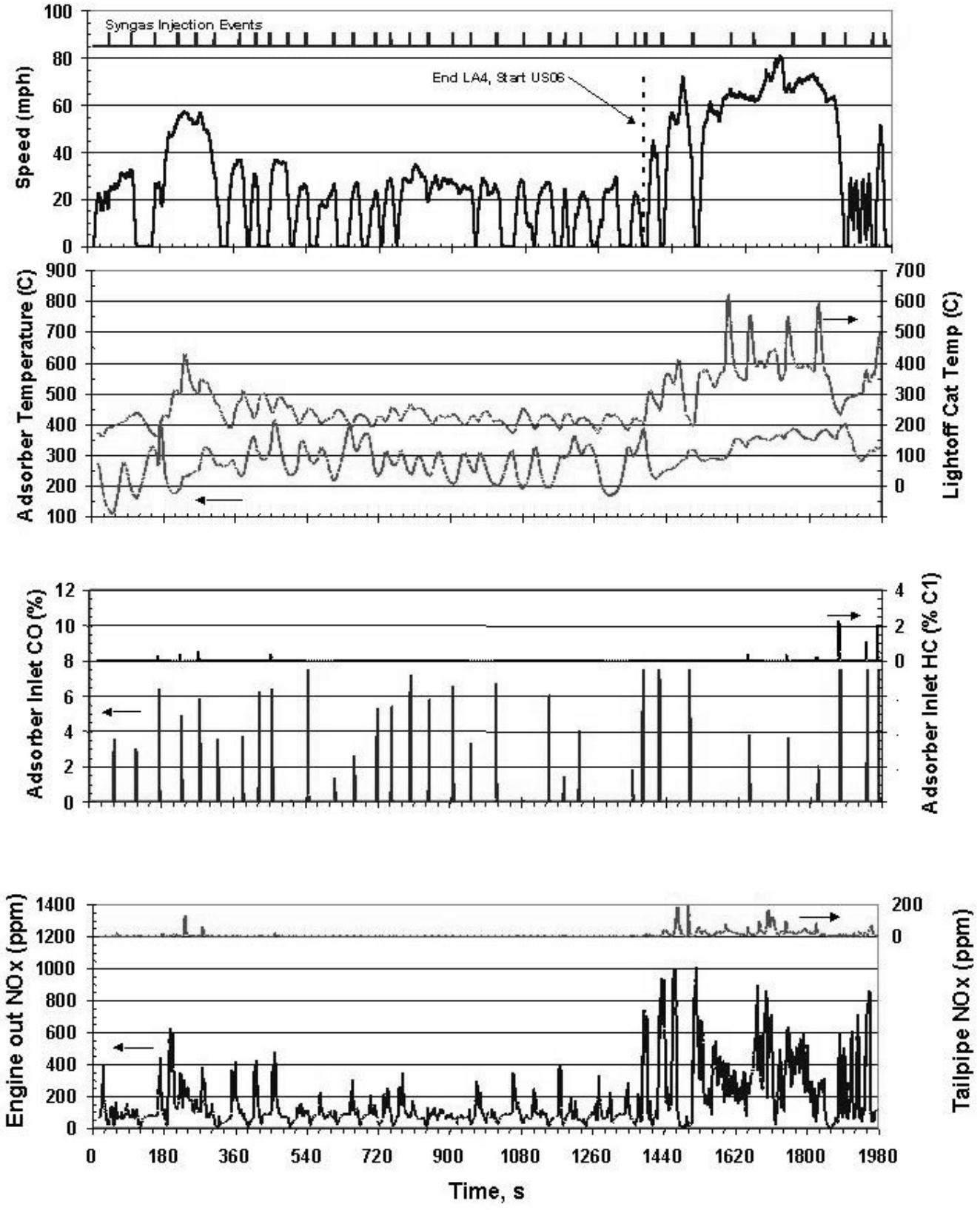

Figure 6. NOx adsorber regeneration data for LA4-US06

Equations 1 and 2 show that regardless of the $CO:H_2$ mole ratio, only one-half mole of oxygen is consumed per mole of synthesis gas. ("Consumption of oxygen" is probably counterintuitive to some, but this terminology is used because the objective of the synthesis gas is to eliminate the oxidizing environment and create a reducing environment.) Equation 3 shows that 1.5 moles of oxygen can be consumed for every mole of carbon from the ethylene in the synthesis gas. Similarly, combustion of diesel fuel will consume nearly 1.5 moles of oxygen per mole of carbon, as shown in equation 4.

Because of the large quantities of excess exhaust gas oxygen, there was a definite perceived advantage to including hydrocarbons in the synthesis gas to reduce syngas demand. Diluting the exhaust gas with larger than necessary volumes of relatively cool gas was obviously not in the best interest of enhancing system performance. Unfortunately, the exhaust temperatures with the 25% ethylene mix were not high enough to promote sufficient oxidation of the HC in the lightoff catalyst. As such, NO_x conversion was poor and HC emissions were high. A few experiments were run to refine the HC concentration to allow some reduction of syngas demand without adversely affecting HC emissions. The gas mix used for all reported NOx adsorber evaluations was nominally 64-66% CO, 31-32% H_2, and 2-5% ethylene. Because syngas demand on the US06 cycle was generally higher than on the FTP, more ethylene was included in these tests. Further reduction of syngas demand is believed achievable with additional optimization of its composition.

Because hydrogen is such a strong reducing agent, a test was conducted with pure CO to examine the effects of including hydrogen. Tests with pure CO resulted in low exhaust temperatures, poor NOx conversion, and high CO emissions on the LA4 cycle. Figure 7 shows the adsorber inlet gas temperature and adsorber bed temperature for the pure CO test run (fine lines) and one of the mixed syngas runs (heavy lines) for the combined LA4-US06. It is clear that oxidation of appropriate levels of hydrogen in the syngas release heat, which thereby promote oxidation of the CO and ethylene upstream of the NO_x adsorber.

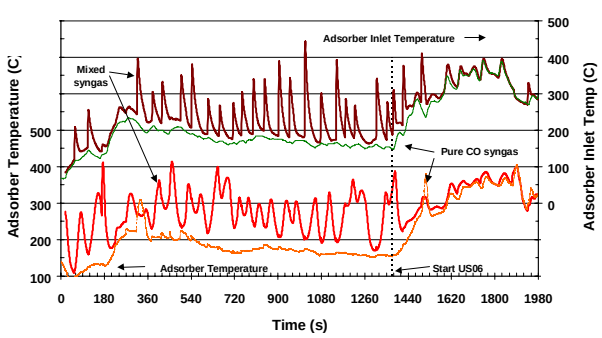

Figure 7. Adsorber temperatures with and without hydrogen in syngas

The adsorber inlet temperature on the LA4 averaged 220C with mixed syngas containing hydrogen, but was below 170C in the pure CO case. The adsorber bed temperature averaged 260-270C on the LA4 with mixed syngas, but only 170C with pure CO. A heated universal exhaust gas oxygen sensor was installed upstream of the NO_x adsorber during all tests, providing information on exhaust oxygen levels entering the NO_x adsorber. With the pure CO syngas, oxygen concentrations remained high during injection, whereas with other syngas mixtures the oxygen was generally depleted to zero. Given these facts, the authors are confident that the hydrogen acted more as a fuel and less as a reductant for NO_x conversion.

RESULTS

The vehicle's baseline emissions were measured with and without the OEM oxidation catalysts installed. The OEM catalysts were observed to provide some NO_x reduction at idle conditions, but drive cycle-resolved NO_x was largely unaffected, as shown in Figures 8-10. The OEM catalysts are effective at oxidizing CO and HC and reducing the total particulate mass by some 30-40%, also shown in Figures 8-10. The effect of fuel sulfur on the factory catalysts was small. Due to the low exhaust temperatures during the light-duty drive cycles, there was very little production of sulfate, even with the 150 ppm sulfur fuel. The small increases in PM with increasing sulfur with the OEM catalysts and the engine out case were largely due to increased soot. This phenomenon was also observed in some of the DECSE results (3). A more thorough discussion of the PM results with the OEM catalysts and the catalyzed diesel particulate filter, including fractionation of the PM, can be found in reference 5.

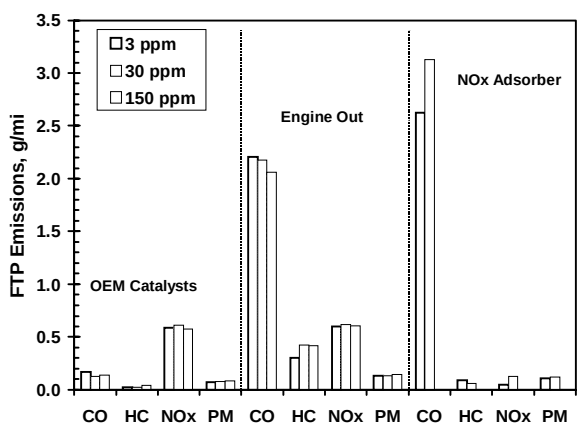

Figure 8. FTP emissions for all evaluations

The NO_x adsorber in conjunction with its close-coupled lightoff catalyst reduced FTP NO_x emissions with the 3 ppm sulfur fuel by more than 90%, from 0.6 g/mi to 0.05 g/mi, as shown in Figure 8. The US06 cycle requires much higher engine loads, and as such generates higher temperatures. While the higher temperature operation

does tend to enhance catalyst performance, the higher space velocities associated with higher load operation can also limit performance somewhat. Figure 6 shows the engine-out and tailpipe NO_x for the LA4-US06. Clearly, the tailpipe NO_x concentrations during the US06 are much higher than on the lighter load FTP. Nonetheless, NO_x reductions of 89% were achieved on the US06 cycle with the 3 ppm sulfur fuel, from 1.3 g/mi to 0.14 g/mi, as shown in Figure 9.

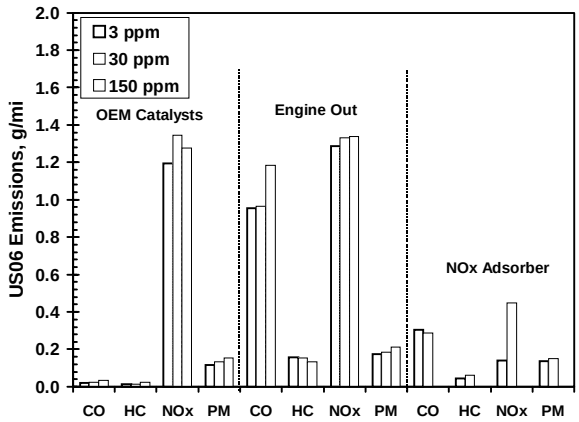

Figure 9. US06 emissions for all evaluations

As noted previously, the Mercedes A170 was not equipped with air conditioning, but the vehicle was tested on the SC03 cycle nonetheless, due to the interest in the different microtransients of the cycle. Baseline NO_x emissions on the SC03 were 0.7 g/mi as shown in Figure 10. With the NO_x adsorber installed, the SC03 emissions were reduced over 95%, to 0.03 g/mi. For these experiments the SC03 was run following a "hot 505." The hot 505 refers to running the first 505 seconds of an FTP, to precondition the vehicle such that engine and catalyst temperatures are always the same before the test.

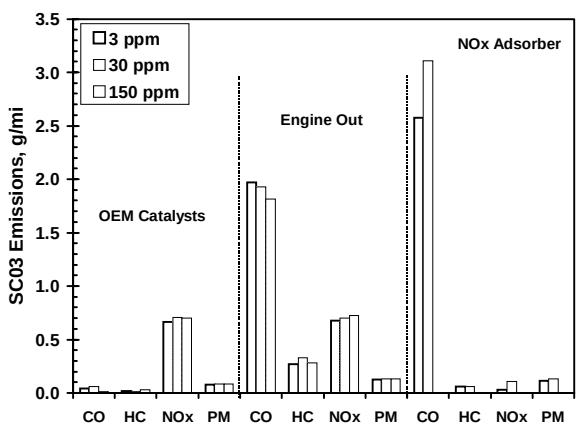

Figure 10. SC03 emissions for all evaluations

SULFUR POISONING – Fuel doped to 150 ppm sulfur was used to simulate short-term sulfur poisoning of the catalyst system. Approximately ten hours or 600 miles of operation on the chassis dynamometer with the 150 ppm fuel were used to load the catalyst system with some 5.7 grams of sulfur, equivalent to 3,000 miles of operation on the 30 ppm fuel. This approach was used because of the obvious difficulties in accumulating thousands of miles on a chassis dynamometer with the adsorber and regeneration system operational. Similar sulfur poisonings were not pursued on the OEM catalysts as these devices have been in commercial use with 300-500 ppm sulfur fuel for a number of years.

After only 3000 miles equivalent aging with 30 ppm fuel, the NO_x reduction was severely impacted. Reduction of NO_x on the FTP went from over 90% to only 80%, as shown in Figure 11. The SC03 cycle (without air conditioning) resulted in similar performance loss, from over 95% to 84%, shown in Figure 12. Because the sulfur poisoning renders some sites in the adsorber catalyst unavailable for NO_x adsorption, the effect is presumably similar to simply reducing catalyst volume, which would effectively increase space velocity. This effect is most apparent in the performance loss on the US06 cycle, with NO_x reduction going from 89% on 3 ppm to only 66% after sulfur poisoning, shown in Figure 13. Assuming that the poisoning effect is linear, one can presume that the system would be less than 50% effective on the FTP after less than 10,000 miles of operation on 30 ppm sulfur fuel. Because complete removal of sulfur from fuel is not likely, it is apparent that methods for desulfurizing the NO_x adsorber may be needed for commercial viability of this technology. Desulfurization (desulfation) is being investigated, but currently requires elevated temperatures over 600C and extended rich operation (8-11,14,15,17,18,21-25), which might prove difficult for diesel engines. An alternative to desulfation could be the use of sulfur traps in the exhaust, which is also being explored (17,26-28).

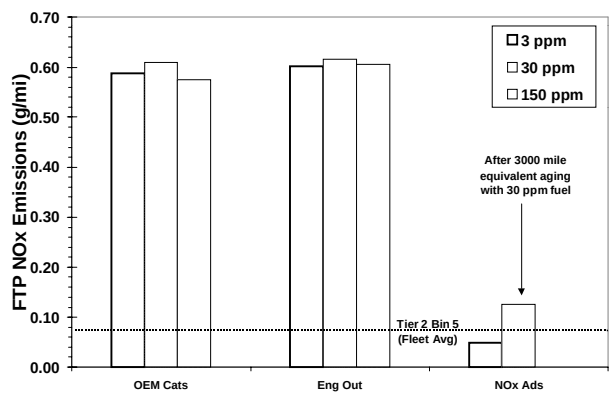

Figure 11. FTP NO_x emissions

FUEL ECONOMY – Fuel economy for the A170 vehicle is shown in Figure 14. The diesel fuel equivalent penalty was determined based on a carbon balance. The fuel penalty associated with regenerating the adsorber with the open-loop system described earlier averaged 11% for the FTP and SC03 cycles, and 3.7% for the US06. This fuel penalty is dominated by the need to deplete exhaust gas oxygen. The theoretical minimum CO needed to

desorb and reduce NO_x is 2.5 moles CO per mole of NO_x. Assuming 100% NO_x reduction, and assuming that all fuel carbon can be converted to CO, the minimum theoretical fuel economy penalty for this vehicle on the FTP would be less than 1%. Because reductant utilization can be on the order of 50% (13), the minimum achievable fuel economy penalty is probably on the order of 2%. Realizing penalties this low will require engine control and regeneration strategies that can lower exhaust gas oxygen without burning excess fuel. One obvious approach to lower exhaust gas oxygen would be to employ inlet throttling during regeneration, which would also add the benefit of reducing the catalyst space velocity.

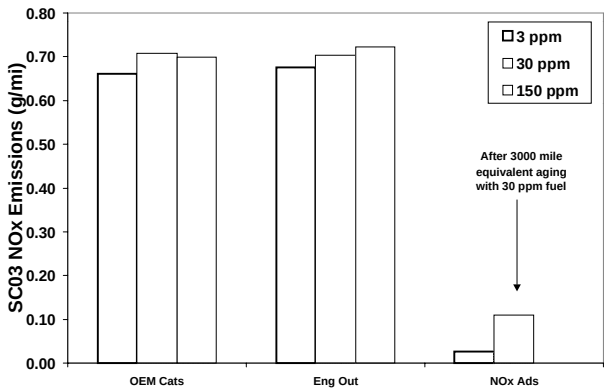

Figure 12. SC03 NO_x emissions

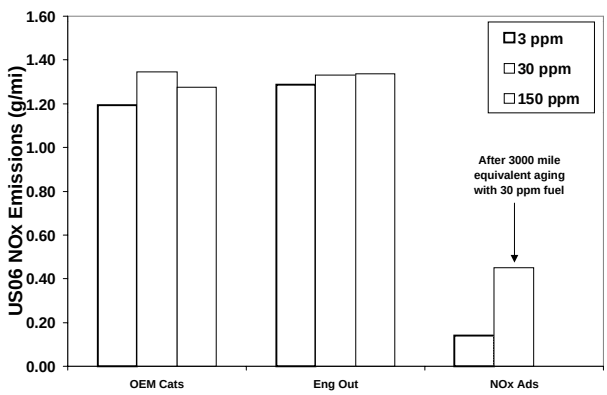

Figure 13. US06 NO_x emissions

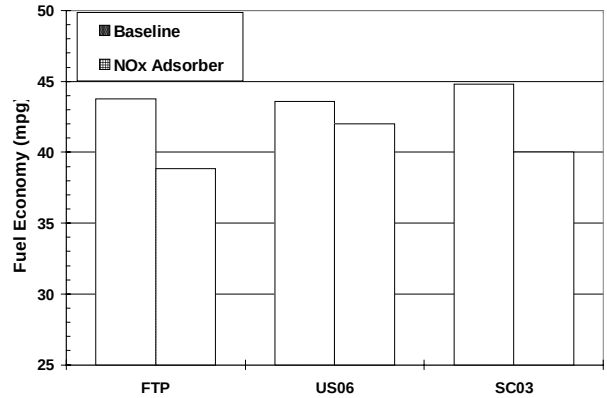

Figure 14. Baseline and NO_x adsorber fuel economy

CONCLUSIONS

- NO_x adsorbers show promise for enabling significant reductions in diesel NOx emissions. NO_x reductions of 92 and 89% have been demonstrated for a light-duty diesel vehicle on the FTP and US06 cycles, respectively, using a synthesis gas injection system to simulate late-cycle, in-cylinder injection of diesel fuel.
- Sulfur loading equivalent to 3000 miles of operation on 30 ppm sulfur fuel caused a marked decrease in NO_x conversion over the FTP and US06, from 92 to 80%, and from 89 to 66%, respectively. Commercial use of the NO_x adsorber in light-duty diesels will require effective desulfurization, sulfur traps, or another solution to the sulfur poisoning problem.
- Diesel fuel equivalent fuel economy penalties of 11 and 3.7% were observed for the FTP and US06 cycles, respectively, for the regeneration strategies used in this program. Fuel economy penalty was dominated by the need to deplete exhaust gas oxygen during regeneration. Inlet throttling may be used to significantly lower exhaust gas oxygen, allowing more acceptable use of fuel. Advancements in engine design and control are key to enabling the NO_x adsorber's use with the diesel engine.

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DEFINITIONS, ACRONYMS, ABBREVIATIONS

CO: Carbon monoxide
CO_2: Carbon dioxide
DECSE: Diesel Emission Control Sulfur Effects
DVECSE: Diesel Vehicle Emission Control Sulfur Effects
DOE: U.S. Department of Energy
EPA: U.S. Environmental Protection Agency
FTP: Federal Test Procedure for light-duty vehicle emissions
HC: Hydrocarbons
LA4: Phases 1 and 2 of the FTP
MECA: Manufacturers of Emission Controls Association
NO_x: Oxides of Nitrogen
OEM: Original equipment manufacturer
PM: Particulate Matter
ppm: parts per million
SC03: Light-duty drive cycle for emissions with air conditioning use
US06: Light-duty drive cycle for high-speed, high-load