ARB Contract # 95-348

Appendices For:

PROTOTYPE DEMONSTRATION OF CHA NOx REMOVAL SYSTEM FOR TREATMENT OF STATIONARY DIESEL ENGINE EXHAUST

Prepared for:
California Air Resources Board and
the **California Environmental Protection Agency**

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May 1998

Appendix A: Task 1: Prototype Design.....1

Appendix B: Task 2: Fabrication of Prototype Device.....12

Appendix C: Task 3: Test Plan and Process Debugging..... 19

Appendix D: Task 4: Start-up and Shakedown Operations.....32

Appendix E: Task 5: Soot Destruction Device Testing.....42

Appendix F: Task 6: Fabricate Regeneration Device and Build Heat Exchanger.....58

Appendix G: Task 7: Test Heat Exchanger and NOx Adsorption.....62

Appendix H: Task 8: Study Gas Residence Time and Particle Size....67

Appendix I Task 9: Demonstration, Technical and Economic Evaluation and Final Report.....70

Appendix A: Task 1: Prototype Design

Work Accomplished

Kick-Off Meeting

The project was initiated through a kick-off meeting held at the CARB offices in Sacramento CA. on October 8, 1996. The purpose of this meeting was to review the work plan, discuss CARB policy on supporting the project. Chang Yul Cha represented CHA Corporation during this meeting. One item discussed was a need to re-organize the project tasks. The original proposal was based on three tasks. These three tasks did not show enough detail to clearly define enough milestones to allow invoicing cycles to be completed.

Revised Project Tasks

The project tasks were revised to better represent milestones. Nine milestones were defined across the eighteen month study period. A list of these project tasks and project schedule follows:

Revision prepared October 15, 1996

The original tasks 1,2 and 3 of our proposal submitted January 12, 1996 and included in our contract dated June 28, 1996 have been expanded here to nine individual tasks without sub-tasks. The purpose of this revision is to structure the project with more clear cut milestones. The work represented by the original Tasks 1,2 and 3 in the referenced proposal/contract has not been changed through this revision. The revised tasks are represented by the following Tasks 1 through 9.

Task 1: Prototype Design
Task 2: Fabrication of Prototype Device
Task 3: Installation and Develop Test Plan
Task 4: Start-up and Shakedown Operations
Task 5: Test Soot Destruction Device
Task 6: Fabricate Regeneration Device and Build Heat Exchanger
Task 7: Test Heat Exchanger and NOx Absorption
Task 8: Study Gas Residence Time and Particle Size
Task 9: Demonstration, Technical & Economic Evaluation and Final Report

Task 1: Prototype Design
During Task 1 a prototype unit will be designed. As a part of this work, the service and engineering specifications for a conceptual commercial unit will be defined. Once these specifications are established, the design of a meaningful prototype test unit can be then developed. A milestone report will be submitted to ICAT at the end of this Task.

Task 2: Fabrication of Prototype Device
Following the completion of the design studies, the prototype device will be constructed and installed into the CHA Corporation laboratory test engine as Task 2. The device will require custom fabrication and assembly. A milestone report will be submitted to ICAT at the end of this Task.

Task 3: Installation and Develop Test Plan
Concurrent with the activities of Task 2, a test plan will be designed as Task 3. The test plan will include an investigation of process and engineering parameters such as flue gas temperature, superficial gas velocity, residence time, regeneration time, and energy consumption. A comprehensive operating manual will also be written under this task. All documentation will be completed before the initial start-up of the prototype device. The completed device will also be installed to our 58 hp diesel test engine as a part of this Task 3. A milestone report will be submitted to ICAT at the end of this Task.

Task 4: Start-up and Shakedown Operations
Following the completion of Tasks 2 and 3, the prototype unit will be tested for any operational problems and the problems will be addressed through adjustments or modifications appropriate for whatever shakedown problems are encountered. A milestone report will be submitted to ICAT at the end of this Task.

Task 5: Test Soot Destruction Device
The soot destruction device is a part of the overall assembly installed during Task 4. A series of tests will be carried out at this time specifically aimed at the performance and regenerating ability of the soot destruction section. Any improvements, modifications or adjustments to this section will be made during this Task 5. A milestone report will be submitted to ICAT at the end of this Task.

Task 6: Fabricate Regeneration Device and Build Heat Exchanger
As a part of the development work, a carbon adsorbent regeneration device will be constructed and tested as part of Task 6. The heat exchanger which will be ultimately needed for the device will be designed and constructed at this point in the project. During initial testing work, the heat exchange system currently installed in our laboratory will be used. This heat exchanger is quite adequate for testing work but is too large to be used in the finished device. A milestone report will be submitted to ICAT at the end of this Task.

Task 7: Test Heat Exchanger and NOx Absorption
The heat exchanger performance and NOx absorption performance will be extensively tested during this Task 7. The unit will be modified as needed to meet any operational problems observed. These modifications will be documented and after each modification. A milestone report will be submitted to ICAT at the end of this Task.

Task 8: Study Gas Residence Time and Particle Size
The effect of residence time and carbon particle will be the focus of Task 8 work. This task will complement the earlier Parametric studies carried out in earlier tasks. The result should be a satisfactory understanding of the influence of each operating parameter. The data collected during each task will be compiled and studied on an ongoing basis. A milestone report will be submitted to ICAT at the end of this Task.

Task 9: Demonstration, Technical & Economic Evaluation and Final Report
The developed device will be transported to McCellan AFB in Sacramento and demonstrated as a part of Task 9 work. This demonstration will serve as the first step in commercializing the developed device. Equally, a detailed technical and economic evaluation will be carried on the developed device. A comprehensive final report will be prepared and submitted at the conclusion of Task 9 which is the conclusion of the project. A milestone report will be submitted to ICAT at the end of this Task.

Schedule and Benchmarks

The schedule is based on an October 15, 1996 project start with a total duration of approximately eighteen months. Each of the above tasks are designed to be carried out during a two month period. At the conclusion of each task a milestone report will be delivered to ICAT as a deliverable for the contract period represented by the report. The schedule and associated benchmarks are shown in Appendix A.

Prototype Design

The design work presented here specifically applies to a 50 hp diesel engine. The conceptual design is applicable to larger applications. The CHA Corporation has been developing the technology represented by this design for the past six years. This experience was combined with input from ultimate commercial users such as the Sacramento Municipal Utility District and the U.S. Air Force. This input was gained through numerous telephone conversations and during a November, 1996 visit to Sacramento by Charlie Carlisle of the CHA Corporation. A copy of a trip report representing this visit is included with this report in Appendix B. The design criterion is centered around two applications. One application is to control emissions from diesel power carts used by the U.S. Air Force. The second application is the control of emissions from standby stationary diesel engine driven electrical generators located in California.

Design Criterion for Power Cart Application

Design criteria for the Air Force application is represented by the following list:

- The existing power carts cannot be modified in any way.
- The NOx abatement unit should be a stand alone, self powered unit on wheels that is no larger than the existing power carts.
- The connections of the exhaust piping from the power cart to the device should be through a simple flexible hose or other means that can be easily disconnected and left behind if the power cart(s) need to be mobilized elsewhere.
- Power is not always available where the power carts are used. If a light cart is used in a given ground operation effort, then 10 amps of 120 volt 60 cycle power is available.
- The device should be designed to operate six months without regular maintenance

Additional notes on Power Carts.

- Power carts all have 12 volt DC starters and battery systems.
- The light carts are powered by a 9.5 hp Deutz single cylinder, air cooled diesel engine, Type RF 121, Version D5746 running at 3,600 rpms. The generator is rated at 7 kW. It produces 120 volt 60 cycle power for two sodium floodlight fixtures that are on a lift boom.
- The power carts produce 400 cycle 24 volt power. They are powered by a 150 hp diesel engine supplied by Cummins, Caterpillar, or Detroit Diesel. Each one is rated at 75 kW.
- The air compressor carts and hydraulic carts are powered by the same engine as the light carts.
- Each power cart is self contained. Fuel tank, connection hoses, connection cables and starter battery are built into each cart.

Design Criterion for Standby Generator Application

- Emissions from unit should not exceed 1.5 $x10^{-4}$ pounds NOx/hour/kW.
- Post treatment emissions from a 250 kW unit not to exceed 0.038 pounds NOx/hour, or 0.076 pounds NOx/hour from a 500 kW unit.
- Maximum NOx removal efficiency to be 90 percent.
- Given the direct cost of diesel fueled energy, the only operation of the unit will be for required tests, maintenance, emergency and Auxiliary Power Program purposes. Total operation less than or equal to 175 hours per year.
- Installation shall be evaluated over the 30 year operating life of the engine.
- A 250 kW rated unit must be devised which will operate for 30 years at a present value cost of $162,900.
- Operation shall be unattended, except for periodic servicing.
- Devise must be able to satisfy regulators that emissions will not exceed guarantees and/or permit limits.
- To minimize cost and operating complexity, the installation should not require continuous emission monitoring equipment.
- Device should fit in existing installation exhaust piping without exceeding engine manufactures back pressure limits.
- Devise should be suitable for outside installation and servicing at either ground or rooftop levels.
- Exhaust cooling feature should be passive and rated to function in no-wind conditions at up to 105 degrees Fahrenheit.
- Device should be Underwriters Laboratories rated for fire, and electrical and microwave safety.

Additional notes on Standby Generators.

- Standby generators range in size from 125 kW to 1000 kW.
- Each generator has a dedicated fuel supply and is housed in a metal sound proof enclosure.
- Starting equipment is 12 volt powered.
- Exhaust is directed through a sound suppresser and released vertically from the unit through a steel exhaust pipe ranging in size from three to twelve inches in diameter.

Process Flow Diagram for Prototype Test System

The 50 horsepower prototype NOx, soot, and VOC abatement device presented here has been designed after considering the above criterion. Figure 1 shows a Process Flow Diagram of the prototype device. Each component is sized to accommodate the exhaust gas output from a 50 horsepower diesel engine. This engine is coupled to a 35 kW three phase 240 volt generator. The engine is loaded by applying generated electrical current to a resistive load bank. The engine is a naturally aspirated, four cylinder, four cycle water cooled unit manufactured by John Deere Company. The model number is 4039 D. This engine produces 100 SCFM of exhaust gas at temperatures ranging from 350 degrees to 1080 degrees Fahrenheit depending on engine load. NOx production is from 150 ppm at no load to 2600 ppm at 100% load. Soot production is 5.6 grams per hour at 50% load. Volatile organic compound (VOC) production is 90 grams per hour at 50% load. The exhaust treatment device is sized for this engine but can be easily fitted to other similar sized diesel engines.

The process flow diagram in Figure 1 shows the flow scheme of both the exhaust gas and carbon adsorbent. Hot exhaust gas leaving the engine is cooled by an air cooled heat exchanger to about 175 degrees Fahrenheit. This cooling is necessary because the carbon adsorbent will not adsorb NOx above 300 degrees. The cooling step lowers the temperature of the exhaust gas enough to promote good NOx adsorption when the gas passes through the carbon filled contactor (B-101). After cooling, the gas is passed through a ceramic soot filter (H-101) where the soot is removed and stored. Periodically, microwave energy (M-101) is introduced to this filter device to selectively oxidize the accumulated soot. Following soot removal, the gas is passed through the carbon filled contactor (B-101) from the bottom to the top where NOx and VOCs are removed and stored. The contactor holds 200 pounds of carbon adsorbent. The cleaned exhaust is vented to the atmosphere through the top of the contactor. As the carbon adsorbent bed becomes saturated with NOx and VOC components a 50 pound portion of carbon adsorbent is removed from the contactor through a dispensing valve (V-101) at the base of the contactor. Simultaneously, 50 pounds of regenerated carbon is added to the top of the carbon bed through valve V-102. The adsorbent in the bottom of the contactor bed becomes saturated with NOx and VOC gases before the adsorbent at the top of the bed. Since gas flow is from bottom to top, the adsorbent at the bottom of the bed is continuously exposed to higher concentrations of NOx and VOCs than the top of the bed. To maintain constant NOx and VOC removal efficiency in the adsorbent bed, fresh adsorbent is added to the top of the bed when saturated adsorbent is removed from the bottom of the bed. After leaving the adsorbent bed, the saturated carbon is regenerated by exposing the carbon to microwave energy. This step converts the NOx and VOCs stored on the adsorbent to carbon dioxide, nitrogen and water. The gaseous products of these conversion reactions are vented to the atmosphere. The carbon adsorbent is cycled and regenerated periodically throughout a given engine run interval. How often this carbon adsorbent is cycled will be one of the parameters investigated during this study.

The exact dimensions and operating principle of each individual piece of equipment in the diagram will be presented in subsequent reports as the various pieces are developed and tested. Generally, all connection piping will be two inch black steel pipe fastened via national pipe threads. Microwave equipment will be 2,450 MHz based with variable power controls. Major equipment is listed in the following Table 1.

Figure 1

CHA Corporation Diesel Exhaust Treatmer Process Flow Diagram

		101	102	103	104	110	111
Phase		Vapor	Vapor	Vapor	Vapor	Solid	Solid
Temperature	F	650	175	170	160	170	200
Pressure	In. H2O	15	13	8	0	0	0
Flowrate	SCFM	100	100	100	100		
	Lb/Hr					50	50

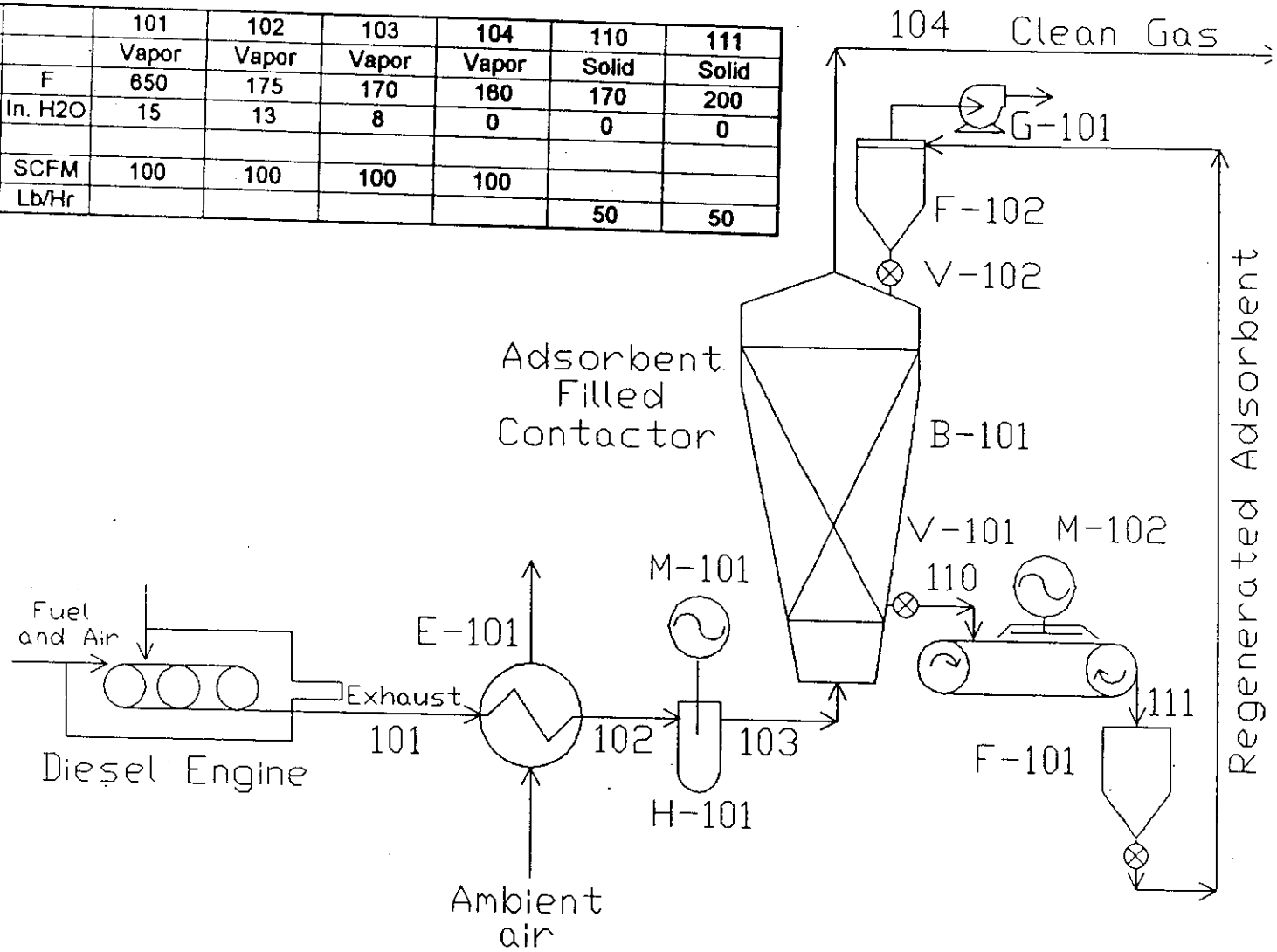


Table 1

Major Equipment List

Identification	Size	Material of Construction	Source
E-101 Air cooled heat exchanger	41,000 BTU/Hr	Galvanized Steel Aluminum Core	Xchanger, Inc. Model AA-250
M-101 Microwave Generator	Variable 0 to 6000 Watts	Steel Cabinet	Cober Electronics, Inc. Model S6F
H-101 Soot Filter	5.625" dia. 6.0" long	Ceramic Monolith	NGK, Southfield, MI.
M-102 Microwave Regeneration Conveyor	8" wide, 6' long.	High Temperature fiberglass/Teflon belt material	CHA Corporation Custom Made
V-101 and V-102 Adsorbent Dispensing Valve	Four inch Star Valve	Steel	Young Industries Muncy, PA.
G-101 Blower	Three inch Blower/Vacuum Pump	Steel with plastic housing	W.W. Grainger Model 6H004
F-102 and F-102 Regenerated Adsorbent Storage Bin	100 pound capacity	Steel	CHA Corporation Custom Made

Adsorber/Contactor Design

Two designs are being considered for the contactor. One shown in Figure 2 is a tapered vertical cylinder five feet tall and thirty inches in diameter on the large end and eighteen inches in diameter in the small end. The taper discourages any wall effects that allow the exhaust gas to bypass the adsorbent. This design will be constructed and tested first. The second design under consideration is a radial flow scheme shown in Figure 3. The radial flow contactor is fifteen inches in diameter and forty eight inches tall. Gas flow is from outside, larger diameter, to inside. The outside housing of the radial contactor is equipped with one inch steel fins that help cool the exhaust gas.

Figure 2

Vertical Contactor

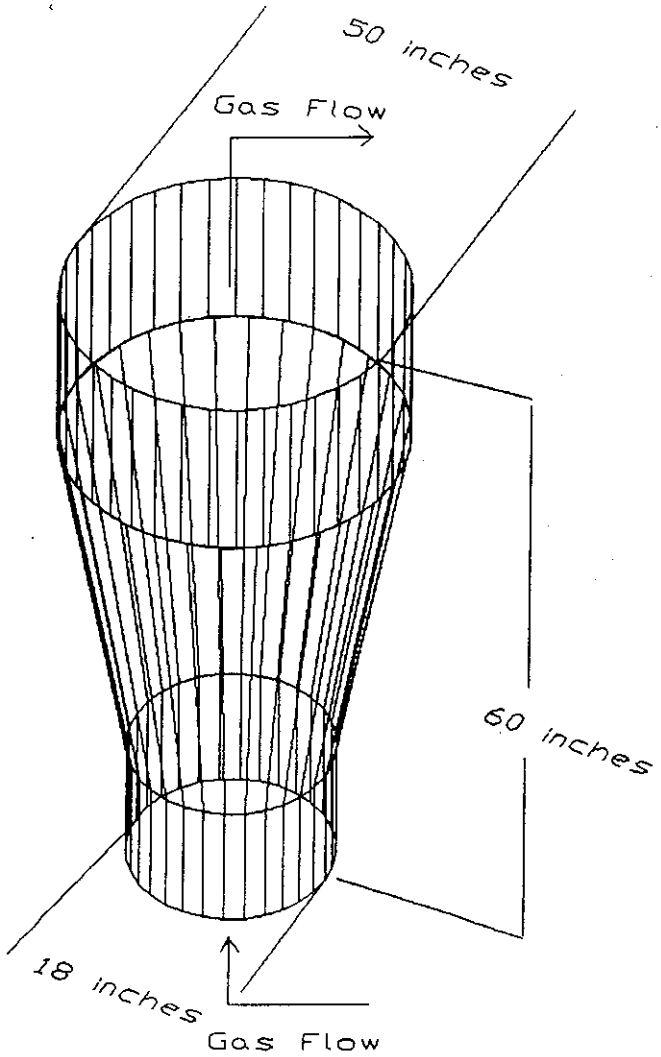
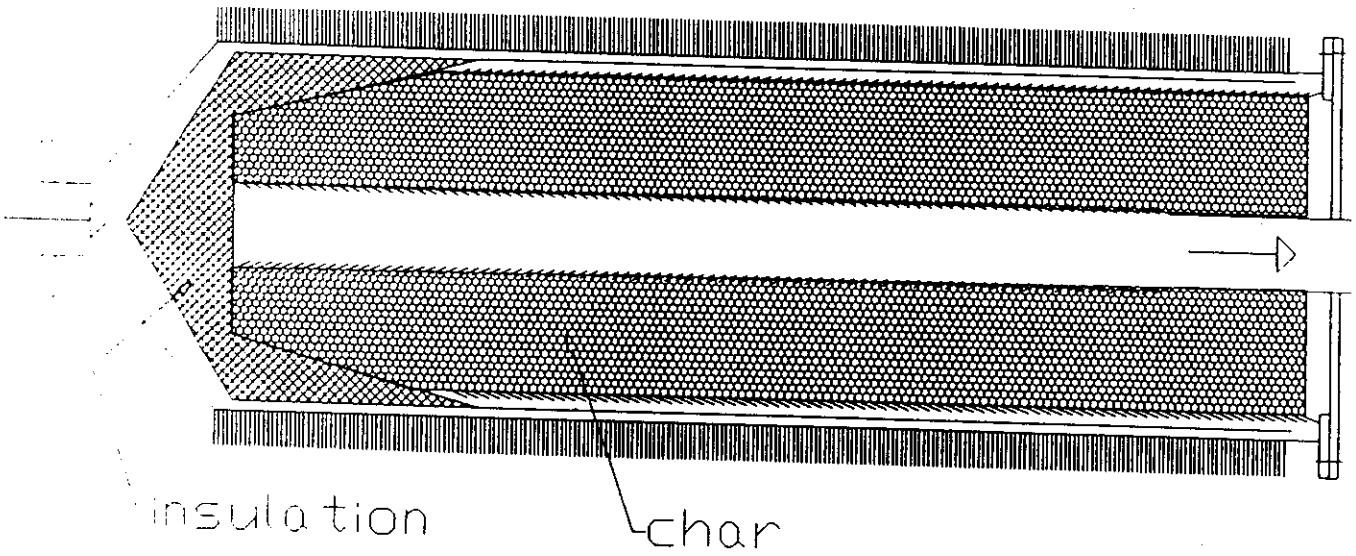


Figure 3

Radial Contactor



CHA NOx ADSORPTION COLUMN

FOR DIESEL ENGINES

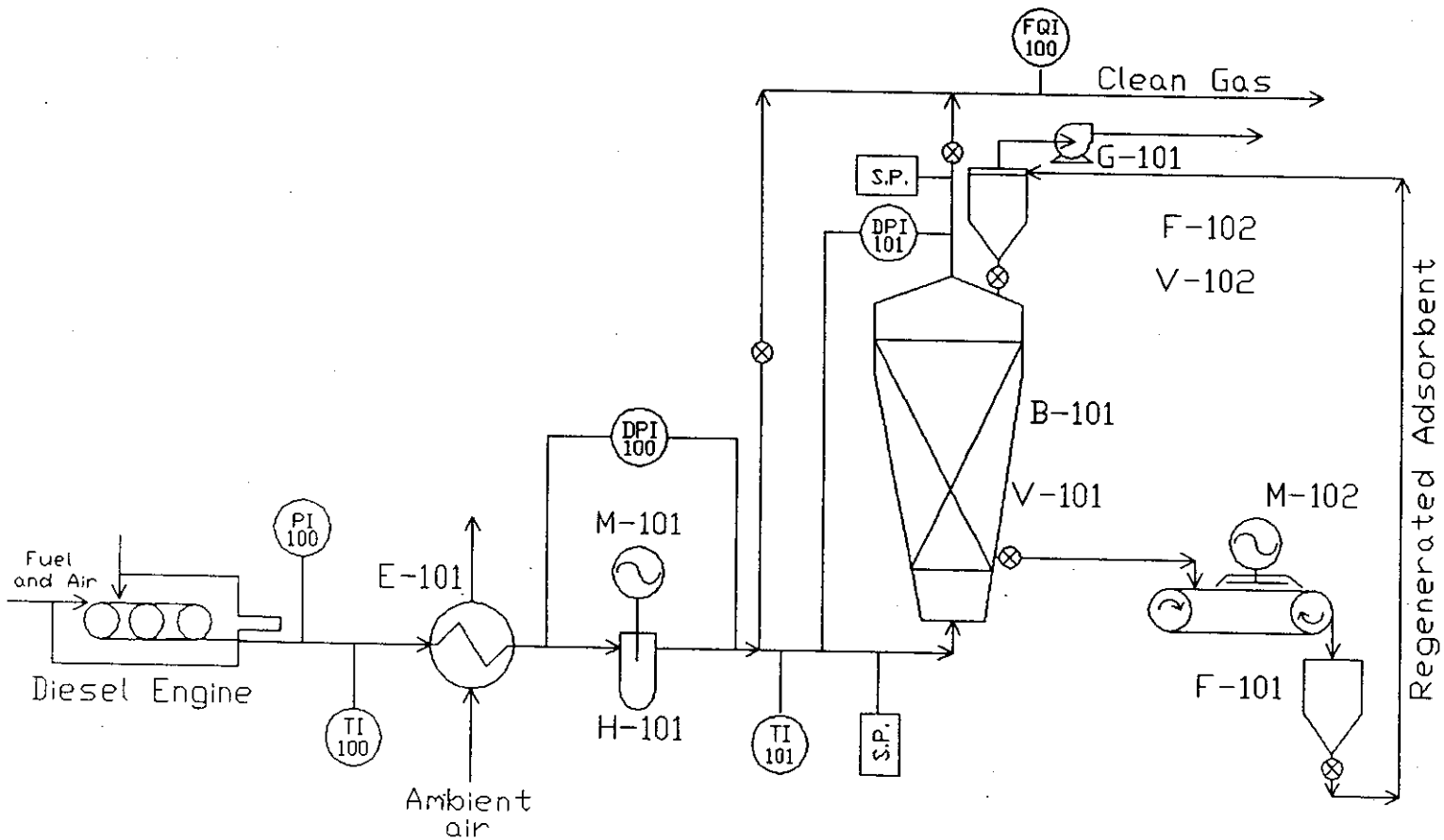
Piping and Instrumentation Diagram

Figure 4 shows the P & ID of the experimental test system. During the upcoming experimental work, Tasks 4-8 and prototype development, the composition of exhaust gases will be measured and recorded using continuous NOx, VOC, CO_2 , CO, and O_2 analyzers connected to a Camile data acquisition system. Exhaust gas temperature, pressure and flow rate will also be continuously measured at various points along the exhaust gas flow path. The process Piping and Instrumentation Diagram (P&ID) shows the location of each of these data acquisition devices. The following is a list of the various instruments noted on the P&ID:

Table 2
Instrumentation List

Instrument Identification	Description
PI 100	Manifold pressure measured by a digital pressure indicator in inches of water.
TI 100	Type J thermocouple reading exhaust temperature near engine manifold in degrees Fahrenheit.
DPI 100	Differential pressure measured across soot filter, inches of water.
TI 101	Type J thermocouple reading exhaust temperature after soot filter, degrees Fahrenheit.
S.P.	Sampling point where a small sample of exhaust gas is directed to the NOx, Oxygen, VOC and CO analyzers.
DPI 101	Differential pressure measured across contactor bed, inches of water.
FQI 100	Vortex gas flow meter measuring exhaust gas flow rate in cubic feet per minute.

Figure 4
CHA Corporation Diesel Exhaust Treatment
Piping and Instrumentation Diagram



Appendix A

Project Start Date; October 15, 1996

Task Identification	10-11,96	12-1,97	2-3, 97	4-5, 97	6-7, 97	8-9, 97	10-11,97	12-1,98	2-3,97
Task 1, Prototype Design									
Task 2; Fabricate Prototype									
Task 3; Install and Develop Test Plan									
Task 4; Start-Up and Shakedown Operations									
Task 5; Test Soot Destruction Device									
Task 6; Fabricate Regeneration Equip. and Heat Exchanger									
Task 7; Test Heat Exchanger and NOx Adsorption									
Task 8; Study Effect of Gas Residence Time and Particle Size									
Task 9; Demonstration, Tec. & Eco. Evaluation, Final Report									

Milestone is Report is Represented by

Appendix B: Task 2: Fabrication of Prototype Device

Fabrication of Prototype Device

This Task 2 work required two months, as scheduled, and comprised construction of the prototype device and installation at our Laramie, Wyoming test laboratory. The device will ultimately be incorporated in a single unit which can be retrofitted to a diesel engine's exhaust system. However, to allow for more thorough testing and modification of the individual components that make up the device, the prototype has been fabricated in a series of individual sub-assemblies.

The purpose of this prototype device is to incorporate the CHA NOx Removal Process in a system that will remove and destroy NOx, VOC, and soot pollutants from the exhaust gas of a 50 horsepower diesel engine. The process utilizes microwave energy to decompose the unwanted pollutants. The decomposition products are carbon dioxide, nitrogen and water. Removal of the NOx and VOC pollutants is accomplished by passing the exhaust gas through a bed of activated coke or char where the pollutants are selectively adsorbed and stored. Removal of the soot is accomplished by passing the exhaust gas through a dedicated soot filter or the char bed can serve as a filter. Once collected, the NOx and VOC pollutants are exposed to microwave energy by passing the carbon adsorbent through a microwave energy field where the pollutants are decomposed. The char or coke adsorbent is re-used after this exposure to microwaves (regeneration). The soot captured by the filter is periodically converted to carbon dioxide by introducing microwave energy to the filter. Microwaves selectively heat the captured soot and accelerate oxidation of the soot without directly heating the filter material.

The process flow diagram is shown in Figure 1. This diagram shows the series of sub-assemblies that have been fabricated and installed in the prototype device. These sub-assemblies are:

1. Diesel engine
2. Exhaust piping
3. Gas cooler
4. Soot filter
5. Adsorbent Filled Contactor
6. Microwave Regeneration Conveyer
7. Adsorbent handling system

The piping and instrumentation diagram is shown in Figure 2. This diagram shows the instrumentation that has been installed to monitor such parameters as gas temperature, gas flow rate, manifold pressure, soot filter differential pressure and contactor differential pressure. Sampling points have also been placed in the gas flow stream at a number of key locations to facilitate exhaust gas analysis. The gas analytical results, pressure and temperature data and gas flow rate data are continuously monitored by a data acquisition system and displayed by a personal computer (PC). Each of these data are recorded and stored in spread sheet format on the PC. Each line of data is marked with the date and time the data is recorded.

Figure 1

CHA Corporation Diesel Exhaust Treatment Process Flow Diagram

		101	102	103	104	110	111
Phase		Vapor	Vapor	Vapor	Vapor	Solid	Solid
Temperature	F	650	175	170	160	170	200
Pressure	In. H2O	15	13	8	0	0	0
Flowrate	SCFM	100	100	100	100		
	Lb/Hr					50	50

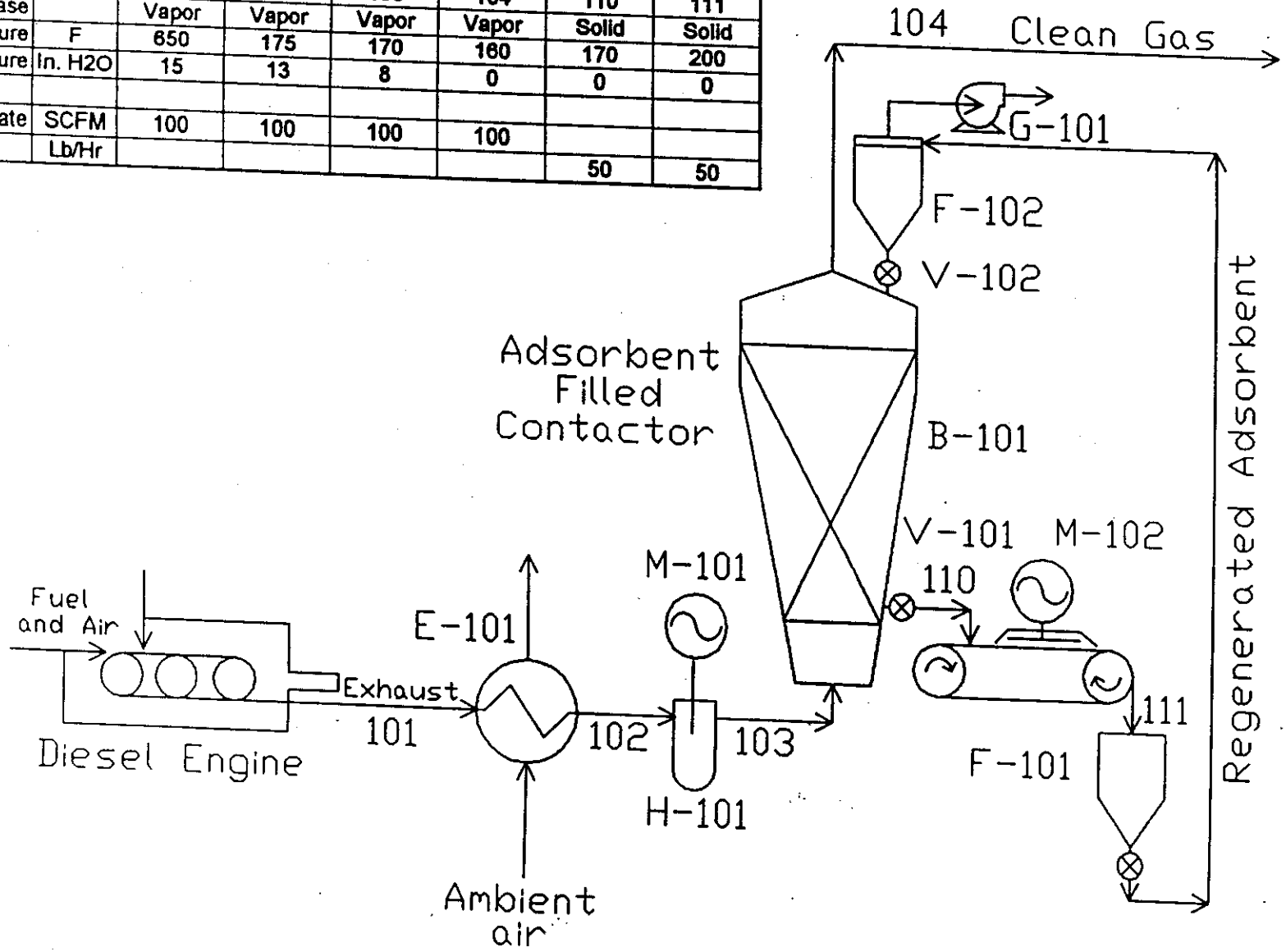
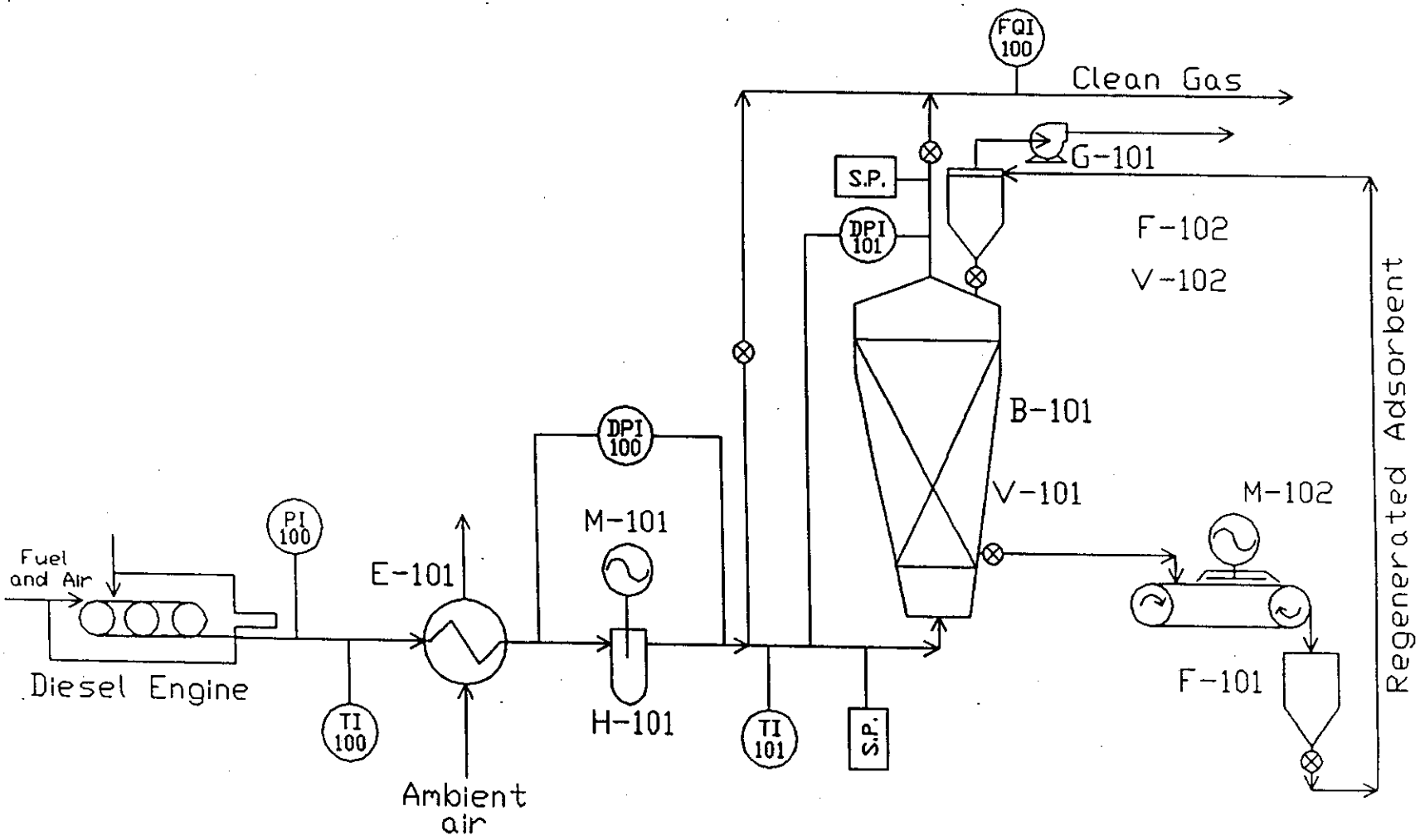


Figure 2
CHA Corporation Diesel Exhaust Treatment
Piping and Instrumentation Diagram

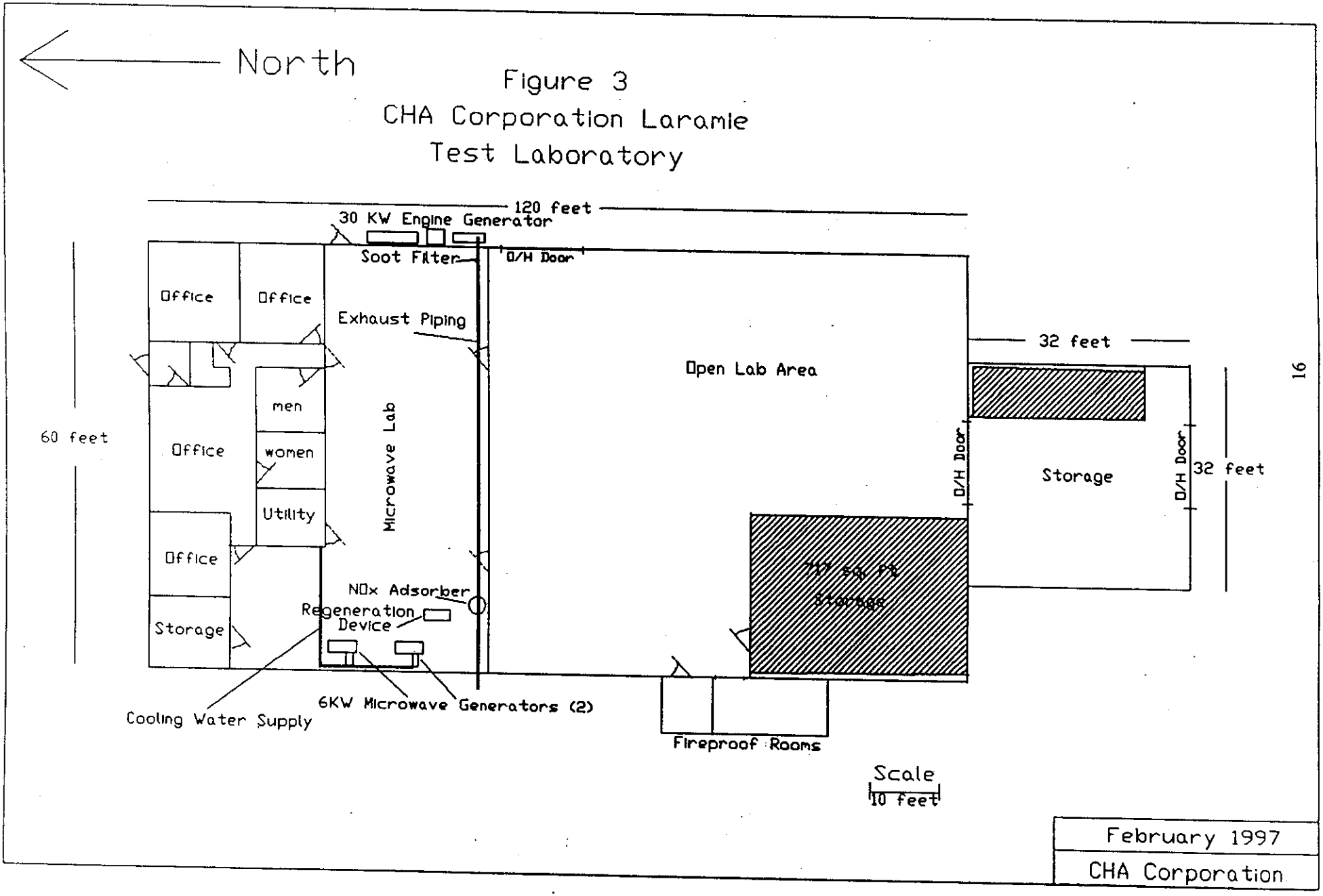


Work Accomplished

Sub-assembly fabrication, piping and instrumentation installation

The sub-assemblies listed above have been fabricated or acquired from suppliers, and installed as a working system (device) in our laboratories. This system is now ready for testing. Figure 3 shows a floor plan of our Laramie laboratories. The system is installed in the Microwave lab. The diesel test engine, fuel tank and load bank have been located outside on the East side of the building. A series of photographs are shown on Figures 4-7 to illustrate the various sub-assemblies. The exhaust manifold of the engine has been adapted to two inch NPT pipe to allow a threaded connection of the engine's exhaust system to black steel pipe. The exhaust gases are routed through the Microwave Lab from East to West and vented to the outside on the West side of the building. Two inch Schedule 40 NPT black steel fittings and pipe were used for the installation. This exhaust pipe is suspended about 10 feet from the Lab floor as it passes through the building. A series of valves and fittings are placed along the exhaust pipe path to allow the gas to be diverted through the soot filter or contactor as needed or bypassed directly to the outside of the building. The exhaust gas piping network is also equipped with the instrumentation listed in the Piping and Instrumentation Diagram shown in Figure 2.

The microwave regeneration conveyer is separate from the gas flow stream and operates as a stand alone unit. Carbon will be manually transferred from the contactor to the regeneration conveyer during the first stages of testing. After regeneration, the carbon will be manually transferred back to the contactor. As the regeneration system becomes more perfected, the transfer of adsorbent will be automated.


North
Figure 3
CHA Corporation Laramie
Test Laboratory
120 feet
30 KW Engine Generator
Soot Filter
O/H Door
Office
Office
Exhaust Piping
Open Lab Area
32 feet
men
women
Utility
Office
Office
Storage
60 feet
Microwave Lab
O/H Door
Storage
O/H Door
32 feet
NOx Adsorber
Regeneration Device
Storage
Cooling Water Supply
6KW Microwave Generators (2)
Fireproof Rooms
Scale
10 feet
February 1997
CHA Corporation

A brief description of the sub-assemblies is offered in the following list:

1. Diesel Test Engine; This engine is coupled to a 35 kW three phase 240 volt generator. The engine is loaded by applying generated electrical current to a resistive load bank. The engine is a naturally aspirated, four cylinder, four cycle water cooled unit manufactured by the John Deere Company. The model number is 4039 D. This engine produces 100 SCFM of exhaust gas at temperatures ranging from 350 degrees to 1080 degrees Fahrenheit depending on engine load. NOx production is from 150 ppm at no load to 2600 ppm at 100% load. Soot production is 5.6 grams per hour at 50% load. Volatile organic compound (VOC) production is 90 grams per hour at 50% load.

2. Exhaust Piping; All piping is two inch Schedule 40, threaded (NPT) black steel pipe sealed with high temperature anti-seize compound. The instrumentation taps and sampling points are accomplished by drilling and tapping the pipe or fittings with 1/4" FNPT at the desired point.

3. Gas Cooler; The exhaust gas is currently cooled by two ten foot lengths of finned tube. This tube is a section of two inch Schedule 40 black steel pipe with a spiraling one inch high and 1/8" thick fin continuously welded to the outside of the pipe. This finned portion of the tube has two wraps per inch of pipe. The twenty foot length cools the exhaust gas from 650 degrees Fahrenheit to 150 degrees Fahrenheit. This finned tube is acceptable in the test lab but will eventually be replaced with a more compact gas cooling unit.

4. Soot Filter; The soot filter is currently a ceramic monolith housed in an aluminum container fitted with microwave coupling taps. Two configurations of this filter will be tested. The ceramic monolith manufactured by NGK will be tested first and a second filter made of Nexcel fibers, a product of 3-M will also be tested. Details on this unit will be presented when the unit is tested and data is made available.

5. Adsorbent Filled Contactor; Two contractors are scheduled to be tested during the study. The first contactor currently installed has been constructed of polypropylene and installed in the exhaust piping system as shown in the Piping and Instrumentation Diagram, Figure 2. The contactor is a tapered vertical cylinder five feet tall and thirty inches in diameter on the large end and eighteen inches in diameter in the small end. The taper discourages any wall effects that allow the exhaust gas to bypass the adsorbent. This contactor was custom made in our laboratories and holds 400 pounds of char adsorbent. Gas is routed from the bottom to the top. A stainless steel support screen holds the carbon 12" above the bottom of the contactor and the carbon is filled to within 12" of the top. The open area on the top and bottom function as a distribution manifold to allow gas to enter and leave the char bed with a reasonably uniform flow distribution. Carbon is added to the contactor through a four inch pipe collar located on top. Removal of spent carbon is accommodated by a similar four inch pipe collar located near the bottom of the contactor and slightly above the support screen. The second contactor design under consideration is a radial flow scheme. The radial flow contactor is fifteen inches in diameter and forty eight inches tall. Gas flow is from outside, larger diameter, to inside. The outside housing of the radial contactor is equipped with one inch steel fins that help cool the exhaust gas.

6. Microwave Regeneration Conveyer; This sub-assembly was custom made in our laboratory. During operation, spent adsorbent, char, is passed under a meanderline microwave coupler on an eight inch wide conveyer belt. The char is about 5/8" deep on the conveyer belt as it passes under the microwave coupler. Belt speed is variable from 0.25 feet/minute to 10 feet per minute. Current settings, 0.6 feet/minute allow for 50 pounds of adsorbent to be regenerated per hour. Microwave energy is from a Cober S6F generator. Power is variable from zero to six thousand Watts. Currently 950 Watts is applied for the 50 pound per hour adsorbent feed rate. This unit will be discussed in more detail as test data becomes available.

7. Adsorbent Handling System; This sub-assembly has not been completed. The char adsorbent is currently transferred manually from the contactor to the regeneration conveyer and after regeneration

the char is manually returned to the top of the contactor. This system will be developed and added after the regeneration system is tested and refined.

Adsorbent Preparation

About 500 pounds of calcined char, provided by FMC Corporation, Kemmerer, Wyoming was received last week. The char was prepared by sieving the material and isolating the 1/4" to 1/2" mesh range. The char was then passed through the microwave regeneration conveyer and exposed to 950 Watts of power for about one minute. The feed rate through the regenerator was 50 pounds per hour. This preparation step, called devolitization, removes most of the low boiling point components in the char and slightly increases the surface area. After devolitization, the char was loaded into the contactor to a level about 12" from the top. The quantity of char loaded was carefully weighed and the weight recorded.

Appendix A

Explanation of Process Flow Diagram Shown in Figure 1

The process flow diagram in Figure 1 shows the flow scheme of both the exhaust gas and carbon adsorbent. Hot exhaust gas leaving the engine is cooled by an air cooled heat exchanger to about 150 degrees Fahrenheit. This cooling is necessary because the carbon adsorbent will not adsorb NOx above 300 degrees. The cooling step lowers the temperature of the exhaust gas enough to promote good NOx adsorption when the gas passes through the carbon filled contactor (B-101). After cooling, the gas is passed through a ceramic soot filter (H-101) where the soot is removed and stored. Periodically, microwave energy (M-101) is introduced to this filter device to selectively oxidize the accumulated soot. Following soot removal, the gas is passed through the carbon filled contactor (B-101) from the bottom to the top where NOx and VOCs are removed and stored. The contactor holds 400 pounds of carbon adsorbent. The cleaned exhaust is vented to the atmosphere through the top of the contactor. As the carbon adsorbent bed becomes saturated with NOx and VOC components a 50 pound portion of carbon adsorbent is removed from the contactor through a dispensing valve (V-101) at the base of the contactor. Simultaneously, 50 pounds of regenerated carbon is added to the top of the carbon bed through valve V-102. The adsorbent in the bottom of the contactor bed becomes saturated with NOx and VOC gases before the adsorbent at the top of the bed. Since gas flow is from bottom to top, the adsorbent at the bottom of the bed is continuously exposed to higher concentrations of NOx and VOCs than the top of the bed. To maintain constant NOx and VOC removal efficiency in the adsorbent bed, fresh adsorbent is added to the top of the bed when saturated adsorbent is removed from the bottom of the bed. After leaving the adsorbent bed, the saturated carbon is regenerated by exposing the carbon to microwave energy. This step converts the NOx and VOCs stored on the adsorbent to carbon dioxide, nitrogen and water. The gaseous products of these conversion reactions are vented to the atmosphere. The carbon adsorbent is cycled and regenerated periodically throughout a given engine run interval.

Appendix C: Task 3: Test Plan and Process Debugging

Test Plan and Process Debugging

Concurrent with the activities of Task 2, a test plan will be designed as Task 3. The test plan will include an investigation of process and engineering parameters such as flue gas temperature, superficial gas velocity, residence time, regeneration time, and energy consumption. A comprehensive operating manual will also be written under this task. All documentation will be completed before the initial start-up of the prototype device. The completed device will also be installed to our 58 hp diesel test engine as a part of this Task 3. A milestone report will be submitted to ICAT at the end of this Task.

Background

The CHA Corporation is currently developing a process to remove NOx, soot, and VOCs from diesel engine exhaust gas. As a part of this development, investigations of current process strengths and limitations are needed. This plan is presented as a guide to these investigations.

The purpose of this project is to incorporate the CHA NOx Removal Process in a system that will remove and destroy NOx, VOC, and soot pollutants from the exhaust of a 58 horsepower diesel engine. Diesel exhaust gas is composed of the following:

Component	Amount
Carbon Monoxide, CO	736 ppm
Nitric Oxide, NO	1287 ppm
VOCs	303 g/h
Soot	1.32 g/hp.h
H_2O	7.20%
Carbon Dioxide, CO_2	7.38%
Oxygen, O_2	9.06%
Nitrogen, N_2	76.10%

Under normal operating conditions, exhaust gas leaves the engine's exhaust manifold at 1080 °F. The process utilizes microwave energy to decompose the unwanted pollutants. The decomposition products are carbon dioxide, nitrogen and water. Removal of the NOx and VOC pollutants is accomplished by passing the exhaust gas through a bed of carbon based adsorbent where the pollutants are selectively adsorbed and stored. Removal of the soot is accomplished by passing the exhaust gas through a dedicated soot filter or the adsorbent bed can serve as a filter. Once collected, the NOx and VOC pollutants are exposed to microwave energy by passing the carbon based adsorbent through a microwave energy field where the pollutants are decomposed. Fresh or regenerated adsorbent is added to the contactor each time adsorbent is removed. The adsorbent is re-used after this exposure to microwaves (regeneration). The soot captured by the filter is periodically converted to carbon dioxide by introducing microwave energy to the filter. Microwaves selectively heat the captured soot and accelerate oxidation of the soot without directly heating the filter material.

The process flow diagram in Figure 1 shows the flow scheme of both the exhaust gas and carbon adsorbent. Hot exhaust gas leaving the engine is cooled by an air cooled heat exchanger to about 150 °F. This cooling is necessary because the carbon adsorbent will not adsorb NOx above 300 °F. The cooling step lowers the temperature of the exhaust gas enough to promote good NOx adsorption when the gas passes through the carbon based adsorbent filled contactor (B-101), but maintains a high enough temperature to prevent condensation. After cooling, the gas is passed through a ceramic soot filter (H-101) where the soot is removed and stored. The contactor holds 400 pounds of carbon adsorbent. The cleaned exhaust is vented to the atmosphere through the top of the contactor. As the adsorbent bed becomes saturated with NOx and VOC components, a 50 pound portion of adsorbent is removed from the contactor through a dispensing

valve (V-101) at the base of the contactor. Simultaneously, 50 pounds of regenerated carbon is added to the top of the carbon bed through valve V-102. Since gas flow is from bottom to top, the adsorbent at the bottom of the bed is continuously exposed to higher concentrations of NOx and VOCs than the top of the bed. The adsorbent in the bottom of the contactor bed becomes saturated with NOx and VOC gases before the adsorbent at the top of the bed. To maintain constant NOx and VOC removal efficiency in the adsorbent bed, fresh adsorbent is added to the top of the bed when saturated adsorbent is removed from the bottom of the bed. After leaving the adsorbent bed, the saturated carbon is regenerated by exposing the carbon based adsorbent to microwave energy. This step converts the NOx and VOCs stored on the adsorbent to carbon dioxide, nitrogen and water. The gaseous products of these conversion reactions are vented to the atmosphere. The carbon based adsorbent is cycled and regenerated periodically throughout a given engine run interval.

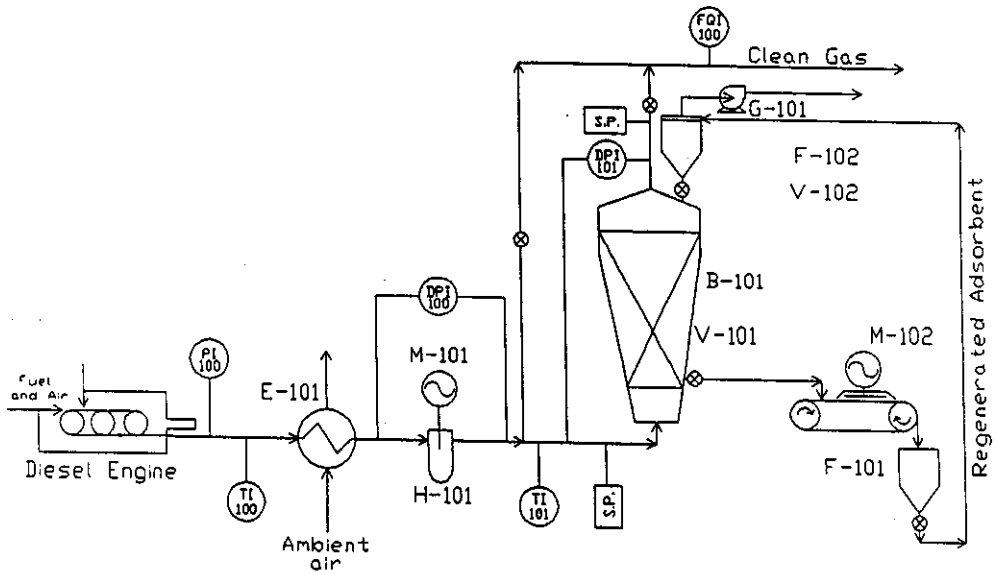


Figure 1: Flow Diagram for CHA NOx Reduction Process

Test Plan

Variable optimization will be based upon a four stage breakdown of the diesel gas cleanup process. The first stage will entail analysis of the soot destruction parameters. The second will involve adsorbent choice. Third, variables influencing adsorption efficiency will be evaluated. Finally, parameters governing the regeneration of the selected carbon based adsorbent will be analyzed.

Soot Destruction

The intended goal of soot destruction testing is to maximize the decomposition of NOx while oxidizing filtered soot in a microwave field. Additional concerns are minimizing pressure drop and energy consumption. Soot destruction is realized through an in-line soot filter in which microwaves periodically induce combustion of the accumulated soot. While this combustion occurs, it may also be possible to utilize the available energy and carbon to decompose some NOx, thereby increasing overall process efficiency.

Experimentation at other institutions has indicated the effectiveness of soot filters, but conventional regeneration practices remain unsatisfactory for prolonged filter usage for diesel engines. Self regenerating filters have had difficulties achieving sufficient temperatures for long enough time periods to combust the accumulated soot. Intermittent short-term operations, typical of portable diesel engines, do not readily allow such conditions. Although diesel engine manifold temperatures can reach 1080 °F after an adequate operating period, temperatures during short intermittent operations do not reach the required 1000°F range needed for soot combustion. Even under optimum conditions, the exhaust gas temperatures will cool significantly before reaching the soot filter. Catalysts have been used to lower the energy requirements for oxidizing the soot. Temperature of 750°F are typically adequate with a catalytic diesel particulate filter. However, even this reduced temperature is difficult to achieve with sporadic engine use and short duty cycles. Unabated soot buildup in the filter eventually creates engine back pressure and poor exhaust elimination. This buildup can lead to poor engine performance or eventual failure. Research investigating the use of electric heaters and fuel burners to provide additional heat and allow complete combustion continues.

The CHA Process provides additional heat and molecular excitation by applying a high energy microwave field to the filtered soot. Soot efficiently absorbs microwave energy and quickly heats up. Consequently, the necessary additional impetus for the combustion of soot under non-ideal circumstances can be provided quickly and efficiently through the application of microwave energy. The CHA Process approach to soot destruction will require considerable testing. A discussion of operational parameters to be varied during the testing follows.

Soot Destruction Variables

During experimental procedures concerning microwave induced soot oxidation, gas flow scheme, filter type, microwave field power density and filter run time will be varied.

Gas Flow Scheme. Two design types are being considered for soot destruction. One applies the microwave energy directly to the filter while continuing process filtration. This technique is similar in concept to the conventional self-regenerating filter, but a microwave field provides the additional energy required for soot oxidation. The second flow scheme has a bypass valve between a pair of parallel fitted filters that will allow one filter to be taken off line while microwave regeneration occurs. This technique lowers energy requirements by eliminating the convective cooling effect of continuous gas flow, but in turn requires more space and initial capitol cost. In addition, periodic microwave regeneration will be tested while continuing filtration.

Filter Type. Two filters types will be tested. A ceramic monolith manufactured by NGK will be tested first, followed by a second filter made of Nextel fibers, a product of 3M. These filters are transparent to microwave field energy. In some experimentation, silicon carbide will be incorporated into the filter design

to enhance the heating rate during the soot destruction period. Silicon carbide molecules efficiently absorb microwave field energy and rapidly heat through intermolecular friction. In addition, silicon carbide does not degrade from the low microwave field intensities utilized within any of our experimentation. This increased heat energy provided by the addition of the silicon carbide will potentially offset the soot density problem that limits the combustion rate within the microwave field. If the soot accumulation on the filter is small, microwave energy produces insufficient intermolecular friction for rapid oxidation.

Microwave Field Power Density. Soot destruction will be performed by bathing the chosen soot laden filter in a microwave field, providing sufficient energy to eliminate the collected soot. Field intensity will be varied to maximize the intended conversion while minimizing power usage.

Filter Run Time. The filter run time indicates the time between filter cleanings through microwave burn-off. A high filter run time will yield a cost benefit trade off between an increased pressure drop over the filter and decreased energy consumption. Diesel engine back pressure is not to exceed 28 inches of water. Filter run time will vary between approximately 30 minutes at the high end and continuous microwave application at the low end. The aforementioned soot density issue may potentially be addressed through longer filter run times.

Experimental Procedure

Testing of soot destruction will be performed under real conditions using exhaust from a loaded 58 hp diesel engine. The exhaust will be channeled through one of the soot filters. The filter is to be surrounded by a microwave helix that will uniformly bath the filter when powered. Field power density and gas velocity will be manually controlled. Two Camile Data Acquisition and Control devices are used in conjunction with 2 PCs to monitor gas flow rate, temperature, and composition throughout each test. Temperature, pressure and concentration data can be taken at various points along the process.

The initial filter run time will be 30 minutes. Initial microwave power will be 900W. Gas velocity will be chosen to provide a residence time beneath that which will provide the soot-oxygen reaction. The velocity will then be reduced until the reactions begin. This will indicate the viable gas velocity range for the process under the experimentally specified conditions. Conditions will be adjusted until a desired velocity range is attained. Power and filter run time will then be optimized for energy and pressure drop. Filter type will be chosen based on ability to remove soot from exhaust gas, duration of filter run time prior to unacceptable pressure drop and facility for regeneration within a microwave field.

Adsorbent

Adsorption is the result of intermolecular forces (primarily Van der Waals) and is similar in effect to vapor condensation. Adsorption occurs at the gas-solid interface. As a result, high adsorbent surface area is critical to adsorbent performance. These heightened surface areas are achieved by creating micropores within the adsorbent through a variety of chemical processes. Carbon based adsorbents are produced by partially combusting a carbonaceous material under conditions that allow uniform reaction. When the solid carbon oxidizes to form carbon dioxide gas, a void is left in the space where the carbon molecule had formerly resided. The accumulation of these voids creates pores that can be relatively uniform in size and distribution with proper care in preparation.

Traditional adsorbent production practices are slow and costly. Uniform reaction requires uniform heating of the carbonaceous material. This demands a slow temperature increase and heightened heat transfer through the application of steam or other medium. Consequently, time and energy demands make adsorbent generation and regeneration expensive under conventional methodology. Microwave fields, however, can be used to uniformly heat the carbonaceous material, allowing for rapid generation and regeneration of the adsorbent. As a result, the CHA process for diesel gas clean-up employs carbon-based adsorbents and microwave energy to take advantage of this quick and cost effective technique.

A variety of carbon based adsorbents will be tested for use with the CHA process. Adsorbent performance significantly influences overall process efficiency. Additionally, adsorbent costs vary greatly. An analysis of adsorbents is therefore integral to any assessment of process efficiency and economic viability.

Adsorbent Types

FMC Form Coke. This adsorbent is the least expensive of those considered. Although the initial surface area and adsorbent capacity are much lower than commercial activated carbon, additional form coke regenerations have been experimentally shown to increase surface areas for over 20 adsorption/regeneration cycles. This testing was performed using simulated gas. Although FMC Coke was an excellent NOx adsorbent for low-temperature gas, it is not clear if this adsorbent will perform well when real diesel engine exhaust gas is used. The diesel exhaust gas contains various components including soot and unburned fuel that were not present during previous experimentation. Coke also has low attrition with respect to commercial activated carbon and is therefore more manageable.

Commercial Activated Carbon. Commercial activated carbon has a much higher initial surface area and adsorption capacity than the FMC form coke. As a result, performance is enhanced, but costs are greater than the FMC form coke. Since the surface areas of the activated carbon are already nearly maximized, regeneration cycles will more rapidly degrade adsorbent efficiency by damaging the pore structure. Consequently, the useful lifetime of the adsorbent is diminished.

Silicon Carbide Based Adsorbent. The final type of carbon based adsorbent is the silicon carbide based adsorbent. Two types will be considered - Alumina and Zeolite. These adsorbents will be coated on particles of silicon carbide. Both Alumina and Zeolite are virtually transparent to microwave field energy, but the silicon carbide heats quickly thereby influencing adsorbent temperature. Major advantages include complete regeneration without loss to consumption and adsorbent flexibility. While the other carbon based adsorbents require fresh adsorbent after each regeneration to offset loss, the silicon carbide based adsorbent are completely regenerated without need for cyclical replacement. Therefore, while the initial investment costs are much higher for this system type, long term costs should be favorable.

Adsorption

After an adsorbent is selected, it will be necessary to determine the optimum performance conditions for that adsorbent. Physical adsorption potential depends on characteristics of both the solid adsorbent and gas passing through the adsorbent. The solid phase must have sufficient surface area to accommodate the adsorption. The gas molecules must have low enough energies to be trapped (by the Van der Waals and polar attractive forces). At higher temperatures, these molecules will have sufficient energy to escape these forces and desorb or avoid adsorption altogether.

In diesel generated exhaust gas, NOx is present only as NO. An important aspect of NOx adsorption and destruction is that the NOx must be in NO_2 form to adsorb on carbon within any reasonable operating temperatures. The combined Van der Waals and polar attraction forces for NO are very small (NO boiling point is -241 °F versus 70 °F for NO_2). Therefore, conversion of NO to NO_2 is critical to pollutant removal. At process temperatures, the conversion reaction maintains equal quantities of NO and NO_2. As the NO_2 is adsorbed to the carbon based adsorbent, the equilibrium of concentration is disturbed, and more NO is converted to NO_2. Therefore, there is a minimum adsorbent bed height for removing all of the NO in the diesel exhaust gas that depends on allowing sufficient time for NO to be converted to NO_2 and followed by adsorption. VOC adsorption is free of any needed chemical reaction and hence can be removed from the diesel exhaust gas with a shorter adsorption bed.

Adsorption Variables

Fundamental variables influencing adsorption optimization goals include exhaust gas temperature, residence time, adsorbent geometry, and adsorbent type. Intended testing ranges or types for these parameters are as follow:

GRAIN SIZE	1 - 10 mesh
PROCESS TYPE	Batch, Continuous
TEMPERATURE	125 - 250 °F
RESIDENCE TIME	1.0 - 3.0 seconds
SUPERFICIAL GAS VELOCITY	25-50 ft/min
GEOMETRY	Radial or Linear

Grain Size. Grain size will be varied between 1 and 10 mesh. Small grain size positively influences adsorption efficiency by decreasing distance between active adsorption sites in the adsorbent. This means that when smaller grain size adsorbent is used, the pollutant has a higher probability at any given point to directly contact the adsorbent. Increased contact is beneficial to adsorption, however, higher pressure drop across the bed occurs for the smaller grain adsorbent.

Process Type. Both batch and continuous processes will be tested. Initially batch reactions will be used for testing primary variables. In the batch experimentation, adsorbent is manually removed from the bottom of the contactor. At the same time, an equal amount of fresh or regenerated adsorbent is added to the top of the contactor. The spent adsorbent is then regenerated and ready to be added back on a subsequent batch cycle. This process type requires external labor and decreases process ease. After determining optimum performance conditions, continuous process schemes will be constructed and tested to increase ease of operation. A continuous system will automatically remove, regenerate and replace adsorbent. A small continuous moving be will be constructed for the test.

Temperature. Inlet exhaust gas temperature is critical to process performance. Under normal operating conditions, the exhaust gas leaves the manifold at 1080 °F. We know from prior work that these types of adsorbents perform best in a temperature range between 125 °F and 250 °F. At the low end of the temperature range, the concern is that the exhaust gas will pass below its water dew point yielding condensation within the contactor. Temperatures above 250 °F decrease the effective capacity of the adsorbent by inhibiting NOx adsorption. Temperature testing will therefore be carried out in this range.

Residence Time. Residence time dictates the amount of contact between adsorbent and exhaust gas. For efficient operation of the adsorbent, sufficient residence time must be allowed. Increased residence time offers diminishing returns with regard to maximizing adsorption. For example, doubling the residence time will not double the NOx adsorption efficiency. Doubling the time may only yield a 10% NOx adsorption improvement. Redoubling the residence time may then only improve performance by an additional 5% or less. In actual fact, with NOx adsorption on activated carbon, residence times yielding nearly 100% adsorption are easily attainable in the 2 to 3 second range. Residence time beyond this threshold may by chosen to increase the breakthrough time of the adsorbent. Breakthrough time is the amount of time before NOx appears in the gas leaving the contactor. If the contactor height is increased beyond that which will provide the necessary residence time for 100% NOx removal, the additional adsorbent will increase NOx breakthrough time. Increased residence time generally requires a decrease in the gas velocity or an increase in available adsorbent. For experimental testing, the superficial gas velocity will be varied between 25-50 ft/min. This velocity, coupled with contactor height, will determine residence time. The desired residence time and gas velocity will be determined experimentally.

Geometry. Radial and linear gas flow patterns will be tested for their respective impacts on residence time, pressure drop, and pollutant adsorption efficiency. Figure 2 depicts the radial flow adsorber. The linear flow contactor, directs exhaust gas from the bottom to the top of the contactor. The bed is tapered outward along the exhaust path to mitigate wall effects in the contactor. This linear flow configuration is illustrated in Figure 3.

Experimental Procedure

Adsorbent screening will be performed using a small moving bed adsorber (4 3/4" Diameter) as shown in Figure 4. Microwave regenerations will be carried out with helix based T-reactor as shown in Figure 5. Diesel exhaust gas will come from our 58 hp diesel test engine. This engine will be run with sufficient load

to form about 1000 ppm NOx in the exhaust gas. This experimental arrangement will allow for consistent testing of the various adsorbents. NOx breakthrough curves will be obtained for each candidate adsorbent to determine the NOx and VOC adsorbing capability of each adsorbent. These curves represent the NOx or VOC concentration of the exhaust gas leaving the adsorbent bed normalized with the corresponding inlet concentration as a function of time. Several cycles of adsorption followed by regeneration will be carried out on a given adsorbent to determine adsorbent longevity.

Figure 2: Radial Flow Adsorption Scheme

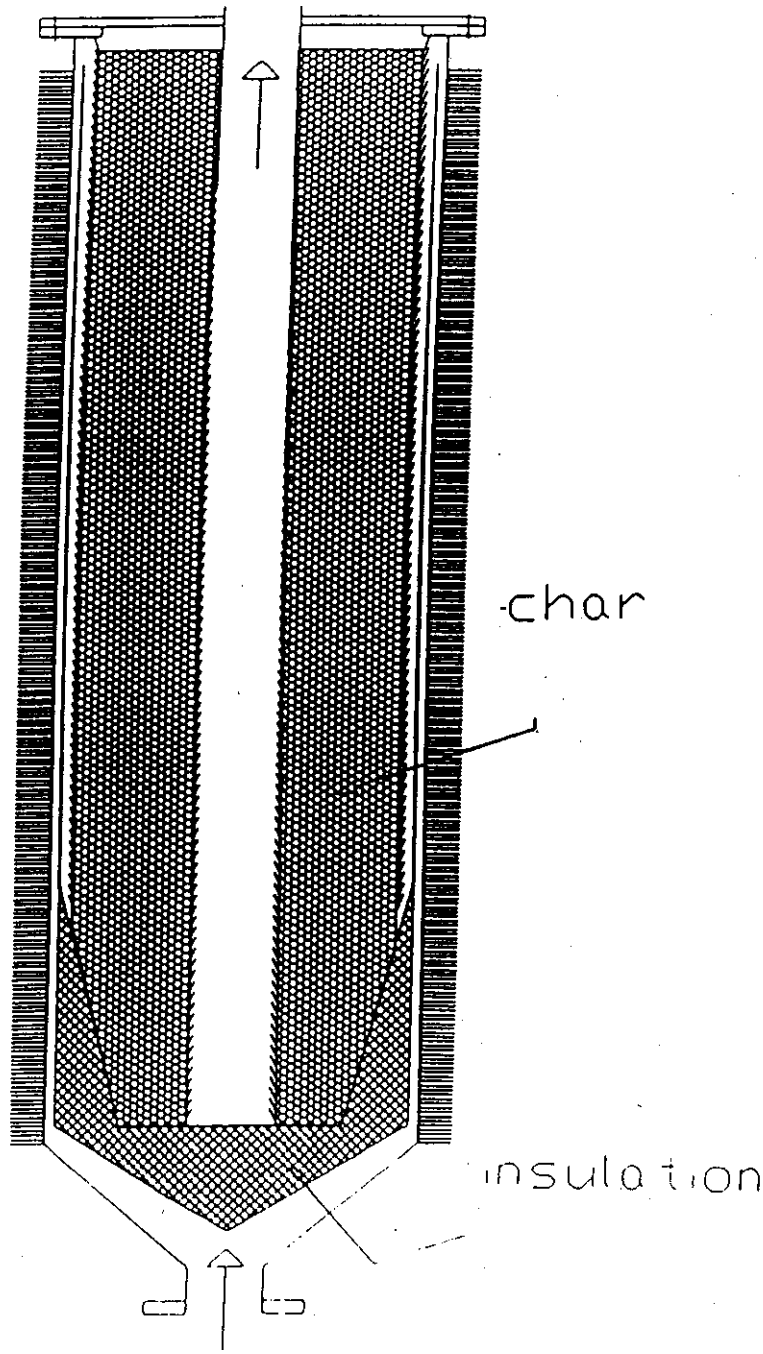


Figure 3 Linear Flow Adsorption Scheme

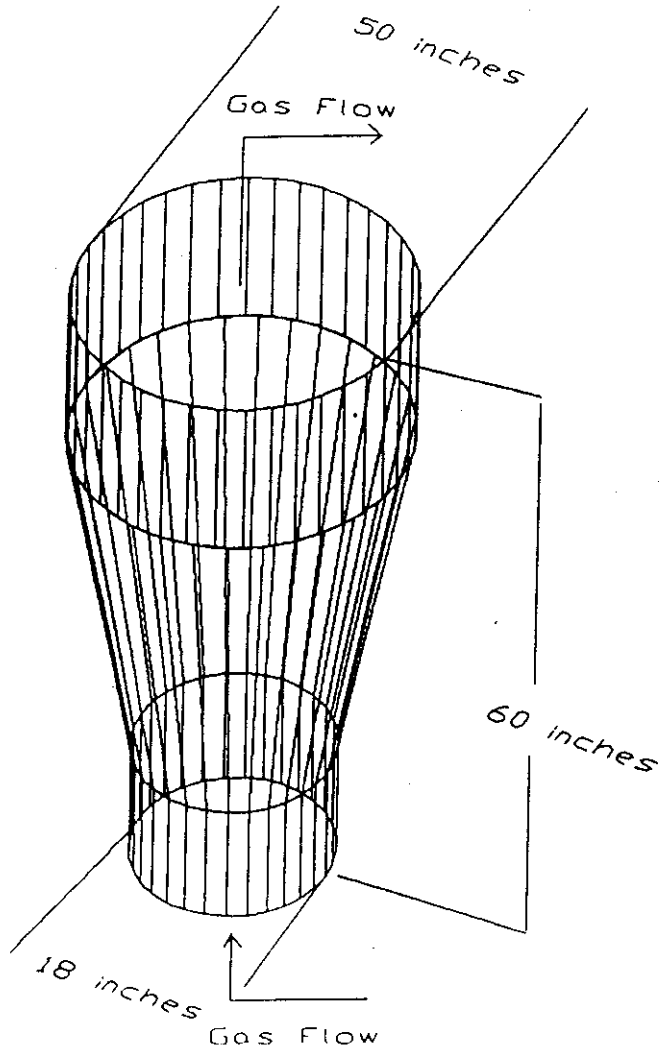


Figure 4: Moving Bed Adsorption Column

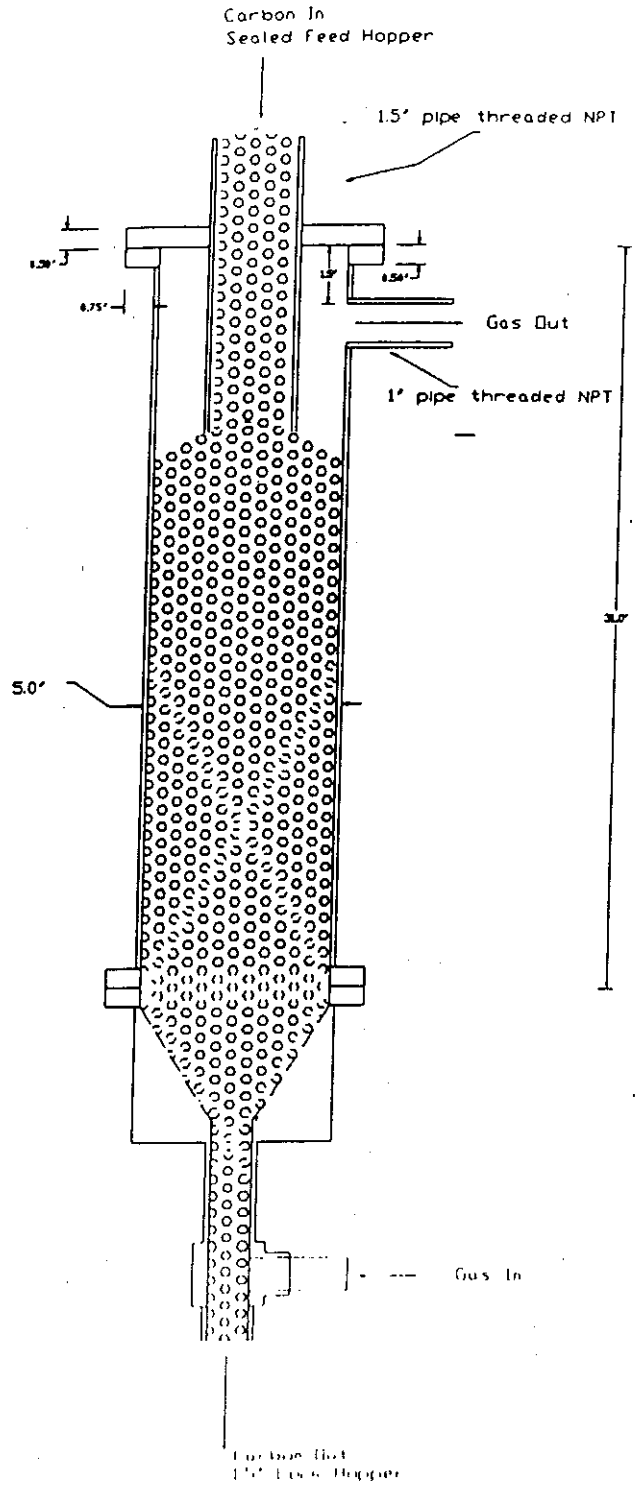
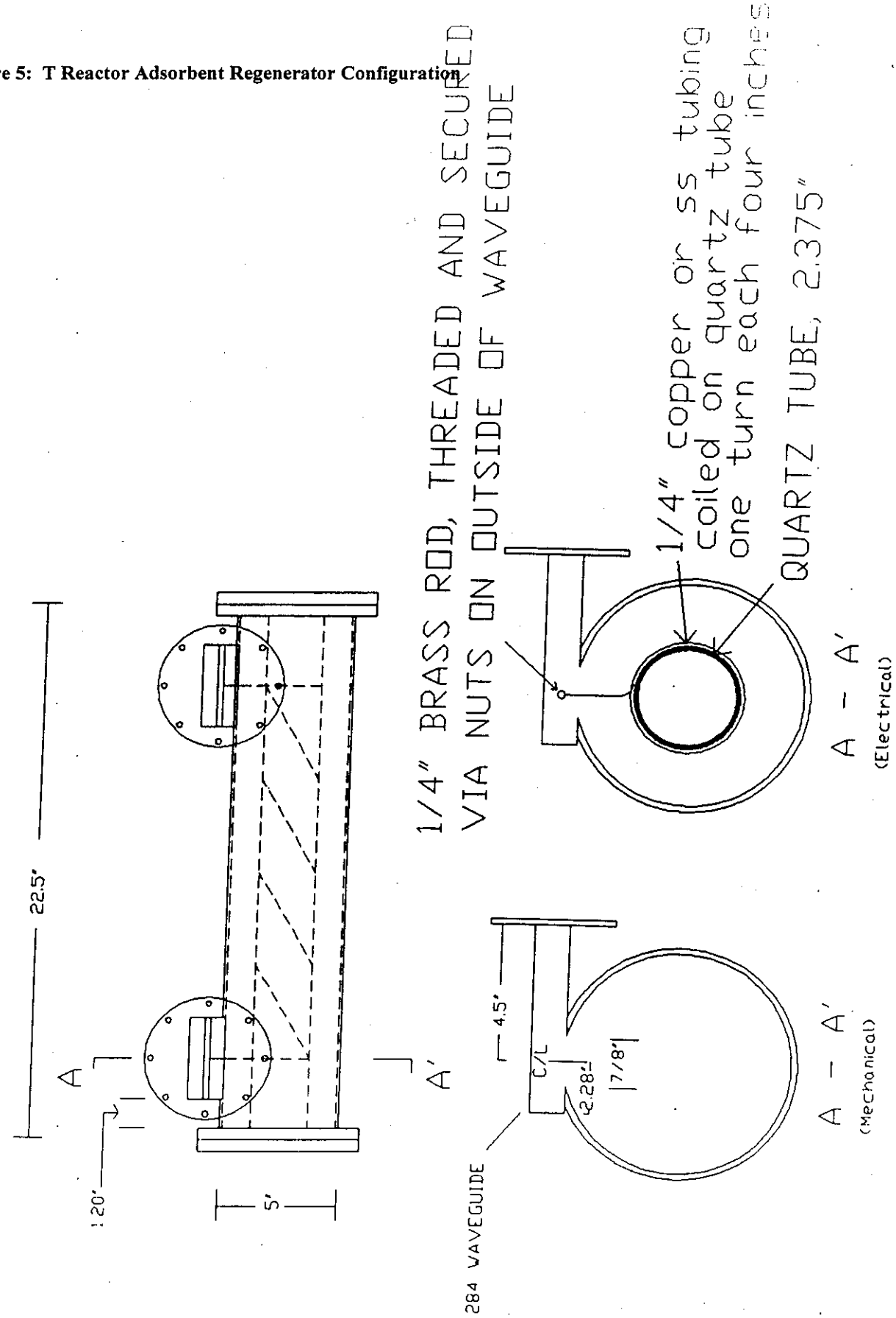


Figure 5: T Reactor Adsorbent Regenerator Configuration



Process Type.- Batch adsorption testing will be carried out in a 2" diameter fixed bed. The continuous adsorber is 4-3/4" in diameter. The batch adsorber will be used as a screening tool and the continuous adsorber will be used to study adsorbent longevity. Batch testing will provide data on adsorbent type and shape only. After deficient adsorbents have been eliminated from consideration, testing will be performed using the small moving bed to determine temperature, residence time, superficial gas velocity and geometry.

Temperature.- The adsorbent temperature will be controlled by adjusting the temperature of the gas entering the adsorber. Temperatures in the range of 125 to 250 degrees Fahrenheit will be investigated.
Residence Time.- Residence time will be varied by simply adjusting the flow rate of the diesel exhaust gas passing through the adsorption bed.
Superficial Gas Velocity.- As residence time is adjusted, as mentioned above, the superficial velocity will follow proportionally. We have found that superficial velocities in the range of 20 to 40 feet per minute are the practical limit of the carbon based adsorbents. The silicon carbide based adsorbents may show a higher tolerance for faster flow rates.
Geometry.- Two flow schemes are under consideration. These schemes are radial and linear flow of the diesel exhaust gas through the adsorption bed. The linear flow regime is much simpler to accommodate experimentally. Base screening of adsorbents and general parametric studies will be carried out using the linear flow scheme. The radial scheme will be incorporated using the best adsorbent on a larger scale.

Regeneration

Carbon based adsorbent regeneration has conventionally been performed by heating the adsorbent using hot nitrogen purge gas or steam until all pollutant molecules are desorbed. The inert gas must be used to avoid combustion of the carbon. Desorbed molecules arethen destroyed or processed in a secondary reactor. In the CHA process, the regeneration procedure employs microwaves to remove and destroy the adsorbed pollutants in one operation.

When NO_2 adsorbed on carbon is exposed to a microwave field, sufficient energy is available for desorption in the same manner as conventional heating. However, the microwave field also provides sufficient energy to trigger parallel NO_2 - carbon reactions of the form:

$$NO_2 + C \rightarrow {}^1/_2\, N_2 + CO_2$$
$$NO + C \rightarrow {}^1/_2\, N_2 + CO$$

Although these reactions are exothermic, they do not produce enough energy to be self sustaining, and are consequently not favored in conventional regeneration. Carbon monoxide produced in the second reaction and remaining VOCs may be refluxed to the soot filter to provide a further reducing agent for incoming NOx. This will reduce adsorbent load and improve process efficiency. VOCs released from the adsorbent will flow through a microwave based catalytic oxidizer and then be converted to CO_2 and H_2O. If the microwave regeneration is performed under a nitrogen purge, carbon will be consumed on a one to one molar basis with NO_2 destruction. Although this carbon consumption will use some of the adsorbent, low to mid grade adsorbents will realize improved surface areas after each regeneration. The carbon consumption effect will eventually lead to micropore collapse and the formation of macropores.

Although the decomposition of NO_2 during regeneration is favorable in a microwave field, some NO_2 molecules desorb and escape before the decomposition reaction can occur. It is therefore necessary in this phase of testing to maximize pollutant destruction while minimizing energy usage and cost.

Regeneration Variables

Power level, adsorbent feed rate and gas purge rate contribute to regeneration efficiency. Proper regeneration requires adequate field strength and conditions favorable to the NO_2 - carbon reaction.

Purge gas treatment must be also be considered. Some adsorbed NO molecules may desorb prior to microwave induced decomposition. These desorbed NO molecules must also be decomposed by secondary treatment, reflux or a combination thereof.

Energy consumption for regenerating the adsorbent will be determined. After adsorbent type, design and regeneration procedure are chosen, the kilowatt energy consumption per pound of adsorbent will be calculated. This consumption calculation dictates the energy expense for the regeneration procedure.

Power Level. Power level for adsorbent regeneration will influence the decomposition efficiency of NOx and VOCs to nitrogen, carbon dioxide and water vapor. Insufficient energy will allow the pollutants to desorb from the adsorbent without destruction, while excess free energy allows potentially unfavorable side and secondary reactions and will result in wasted energy.

Adsorbent Feed Rate. This aspect is of particular importance for continuous regeneration. Sufficient adsorbent residence time must be allowed for complete destruction of the adsorbed pollutants. Bed depth must be kept below effective field penetration depth.

Gas Purge Rate. The regeneration phase will be purged with nitrogen gas. Nitrogen is used to avoid potential combustion of the carbon based absorbent beyond that needed for pollutant destruction. If the N_2 flow rate is too fast, the adsorbent will be cooled through convective heat transfer, requiring further energy expenditure. Additionally, the residence time of the pollutants within the carbon matrix after desorption will be diminished. This will increase the NOx and VOC content in the exiting purge gas by diminishing the opportunity for decomposition within the matrix.

Purge Gas Treatment. The purge gas leaving the regeneration process will contain small amounts of NOx that desorbed and left the carbon adsorbent matrix without reacting as well as carbon monoxide produced from carbon NOx reaction. This leads to the necessity for secondary treatment of the purge gas. Either the purge gas can be sent back to the contactor, or a second stage microwave based reactor can be added that contains a Rh/Pt catalyst to reduce the remaining NOx. This catalyst effectively destroys NOx traces and oxidizes carbon monoxide in gases containing very low oxygen concentrations. Since the purge gas is nitrogen and oxygen from the initial exhaust is spent during regeneration, the Rh/Pt catalyst will effectively destroy the NOx remaining in the purge gas. Several purge gas treatment configurations involving these treatment methods will be examined. In addition, VOCs released from the adsorbent need to be destroyed in the catalyst bed.

Experimental Procedure

We currently have two working devices for regenerating the adsorbent using microwave energy. These devices are the meanderline with a belt conveyor and the helix based T reactor where the material passes vertically through the reactor via a lock hopper. The meanderline is capable of regenerating 100 pounds per hour and requires a minimum load of 10 pounds for operation. The helix configuration can regenerate 10 pounds per hour with a single batch regeneration of about 2 pounds. The helix is therefore more suited to parametric experiments where small scale testing will be performed, while the meanderline is appropriate for larger scale use. Figures 5 and 6 show these two regeneration devices. Each of these reactors do a good job in efficiently coupling microwaves to the saturated adsorber.

Data Analysis

Experimental data from each aspect of the testing will be analyzed to isolate critical parameters. Primary variables will be determined through bench scale testing before moving to integrated process testing.

Soot destruction performance data will be collected over the range of testing parameters and used to choose which filter design will be most beneficial to process efficiency.

1. Adsorbent data will first be collected through batch experimentation for the purpose of screening adsorbent types. After fundamental variable ranges for the most promising adsorbents are determined, moving bed experimental data will be taken. Temperature, residence time, and superficial gas velocity will be systematically varied to determine variable impact on performance. Analysis of this data will provide information regarding the direct and interactive impacts of the variable set. This information will then provide the parameters need to design an optimized adsorption system.

2. Regeneration efficiency will be measured by examining performance of regenerated samples, BET surface area results and weight loss data. By varying the effective parameters of the experimentation, optimized regeneration configurations may be obtained. Energy and surface area data representing full regeneration potential will be obtained by use of the T reactor regenerator. When purged with nitrogen at 1000 Watts, this configuration rapidly regenerates adsorbent. This data may then be compared as a baseline to the continuous belt/hopper configuration as a performance guideline. Adsorbent feed rate and power level for the continuous regenerator will then be calculated according to the batch reactor guidelines.

After the three independent levels of data analysis are completed, the integration of sub-processes may be undertaken. By initially breaking down the process and isolating primary and interactive variable effects, unification will be more readily facilitated. Upon implementation of the process complete, parameters will be fine tuned to provide the most efficient pollutant destruction performance.

Appendix D: Task 4: Start-up and Shakedown Operations

Adsorption Column Shakedown Testing

The adsorption column as initially presented in the prototype design (Task 1) was a semi-batch contactor containing 200 pounds of carbon adsorbent. The diesel exhaust gas stream flowed from bottom to top through the contactor. As the carbon adsorbent became saturated with NOx and VOC components, a 50 pound portion of fresh carbon adsorbent was added to the top of the column while an equal portion of saturated carbon was removed from the bottom. After initial testing, the adsorbent mass was increased to 600 pounds consequently increasing the contactor volume. While the design configuration of this process proved capable of sufficiently adsorbing the pollutants from the diesel exhaust gas, the large size and mass made the system design awkward for a portable continuous system. To mitigate the size problem, a small moving bed was fabricated and used to determine parameters for a more compact contactor design.

Carbon Adsorbent Performance Testing

Batch experiments for adsorbent screening were carried out to isolate candidates for use in moving bed efficiency testing. Adsorbent selection strongly influences process adsorption capacity and rate, as well as cost.

Adsorbent Testing Procedure

Five adsorbents were analyzed for process suitability. Each adsorbent was tested by passing 35 SCFH of raw diesel exhaust gas through a 24-inch bed of the adsorbent contained in a 2-inch ID tube. Inlet and outlet NOx concentrations were recorded over the course of the saturations to determine breakthrough times and adsorption capacity. Adsorbent types, sizes and sample weights were as follow:

Adsorbent	Size	Sample Weight, grams
FMC Form Coke	<1/4"	586.4
FMC Form Coke	10 mesh	652.0
FMC Calcined Char	10 mesh	612.5
FMC Calcined Char	10/20 mesh	676.0
Granulated Activated Carbon (GAC)	8/16 mesh	639.5

Table 1: Adsorbent Types for Sample Testing

Adsorbent saturation was continued until outlet NOx concentration was equal to 80% of the average inlet concentration. Adsorption capacity calculations therefore reflect saturation values slightly lower than possible for the given average inlet concentrations, but sufficient for sample comparison.

Adsorbent Testing Results

The NOx breakthrough testing results suggest two possible adsorbents for use in the NOx adsorption column. FMC Calcined Char (10 mesh) and Granulated Activated Carbon (8/16) performed more effectively than the other sample types. Breakthrough curves for these tests are presented in Appendix 1. The following table describes respective performances.

Adsorbent	Size	NOx Inlet Average (ppm)	20% Breakthrough Time (hrs)	Adsorption Capacity (g/100g)
FMC Form Coke	<1/4"	638	2.72	1.47
FMC Form Coke	10 mesh	622	2.16	0.51
FMC Calcined Char	10 mesh	661	4.03	1.39
FMC Calcined Char	10/20 mesh	719	0.85	0.77
Granulated Activated Carbon (GAC)	8/16 mesh	716	8.04	2.72

Table 2: Adsorbent Performance Comparison

The amount of NOx adsorbed per 100 grams of carbon adsorbent (adsorption capacity) was calculated by integrating NOx adsorption over the saturation period until 80% of inlet concentration was achieved. The capacity as indicated represents the number of grams of NOx adsorbed per 100 grams of adsorbent. GAC performed best at 2.72 g/ 100g, while FMC Form Coke (<1/4") and FMC Calcined Char (10 mesh) performed at slightly better than half of the GAC capacity. The remaining two sample types performed poorly in comparison.

The 20% breakthrough time indicates the time for the outlet gas NOx concentration to reach 20% of the inlet NOx concentration. Only the GAC and Char (10 mesh) samples performed adequately in this area at 8 and 4 hours respectively. Although the GAC efficiency is nearly twice that of the 10 mesh char in both measured categories, it is important to note that the GAC costs over 10 times more than the char making char a potentially cost effective candidate.

Small Moving Bed Design and Operation

A 5-inch moving bed was designed and constructed immediately prior to the Task 3 milestone report. Over the past 2 months, parametric testing using this moving bed has provided information useful in designing a smaller prototype contactor to replace the large semi-batch system. The 5-inch moving bed is depicted in Figure 1. The moving bed test configuration allows bed height, carbon adsorbent repletion rate and diesel exhaust gas flow to be manipulated. Lock hopper actuation time controls the adsorbent repletion by automatically dumping 110 grams of saturated adsorbent out the bottom of the bed while introducing 110 grams of fresh carbon at the top. The actuation time was adjusted to control the repletion rate. By adjusting these three parameters and recording adsorption efficiency at steady state, the important parameters could be isolated for consideration in the prototype refinement.

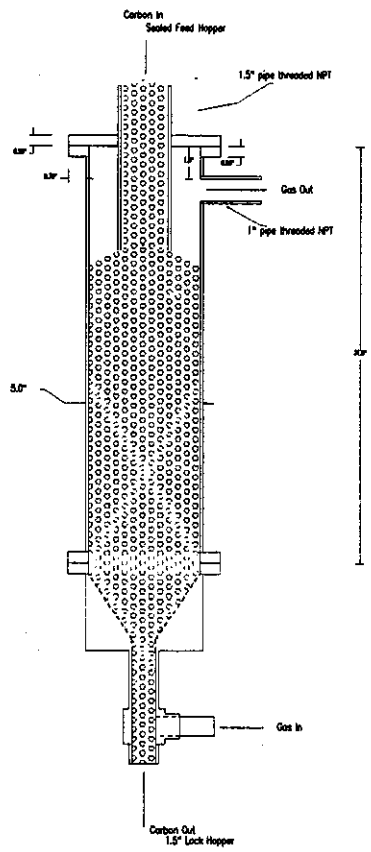


Figure 6: 5-inch Moving Bed Design

Experimental Design

Moving bed parametric testing was performed for the following experimental conditions:

Bed Height	12, 17 and 22.5 inches
Gas Flow Rate	2, 3 and 4 CFM
Actuation Time	5 - 30 minutes

By varying these parameters, a variety of residence times, adsorbent linear velocities and gas space velocities were achieved. Regression analysis was performed to determine variable importance. All testing was performed using raw diesel exhaust gas from our 58 hp diesel engine loaded to produce approximately 700 ppm NOx. The diesel exhaust gas was pre-screened to remove large soot particulate matter. Average reduction efficiency was determined using the arithmetic mean of the peak and minimum reduction efficiencies after the adsorption column stabilized for the given test conditions. The maximum efficiency reflects the adsorption rate achieved immediately following adsorbent repletion actuation. The minimum efficiency represents the efficiency prior to actuation.

A full range of experimentation was run using granulated activated carbon. A selected range of further experiments is being performed on FMC char to determine the relationship between the char and GAC results.

Experimental Results

The results of the moving bed adsorption efficiency testing with granulated activated carbon are depicted in Figure 2. The data is displayed with data point symbols differentiating bed height and best fit line shading corresponding to diesel exhaust flow rate. These results indicate the expected trend of improved efficiency with increased dimensionless mass flow rate. The dimensionless mass flow rate is calculated by dividing the rate of carbon adsorbent repletion in grams of carbon per hour by the NOx throughput, also in grams per hour. This dimensionless group allows for scalable comparisons when considering larger column designs.

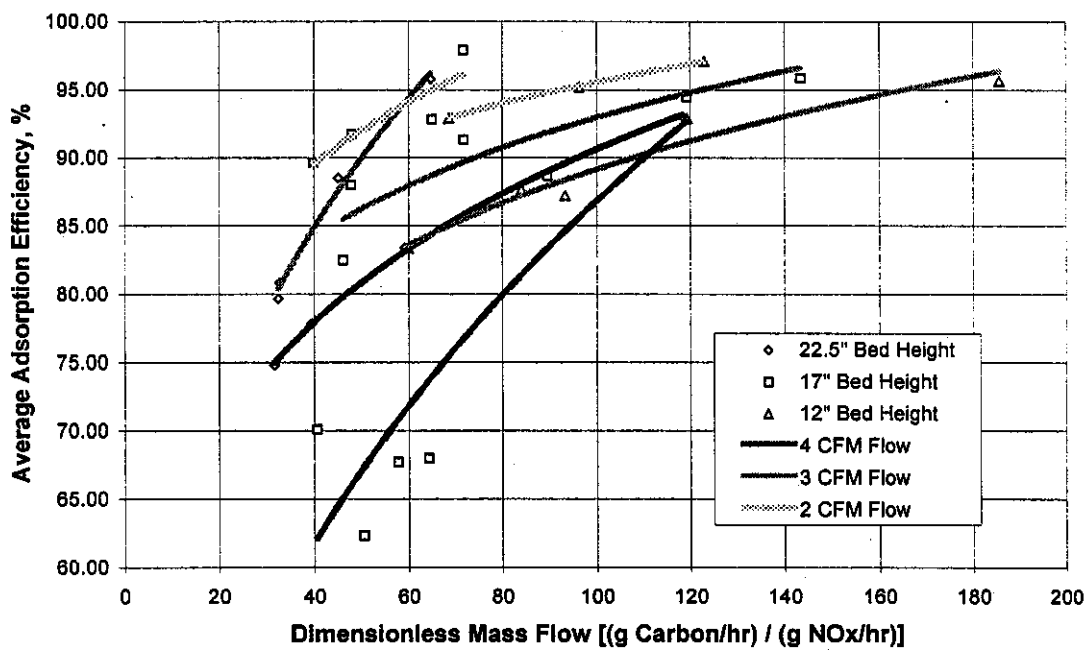


Figure 7: 5-inch Moving Bed Adsorption Efficiency for Granulated Activated Carbon

The importance of the dimensionless mass flow rate validates the feasibility of a reduced size moving bed prototype. By providing a high adsorbent repletion rate, the dimensionless mass flow rate can be kept

above 100 thereby allowing for a high average efficiency without the large contactor volume that was needed for the original design. A smaller adsorber will improve the portability of the process.

Bed height and linear velocity were isolated to determine their respective impacts on moving bed efficiency. Figure 3 illustrates the effect bed height has on efficiency for a series of comparable data. The data points illustrated had identical carbon repletion rates (440 g/hr) and were graphed by their performance as a function of bed height. Data is plotted without regard to exhaust flow rate, so deviation within a given bed height is due to different NOx throughput and velocity.

Results indicate that the efficiency of the moving bed column, assuming a reasonable adsorbent repletion rate for a given exhaust flow rate, is independent of bed height as long as a minimum bed height is achieved. This further justifies the smaller moving bed scheme for the improved prototype design.

An analysis of the variance of adsorption efficiency by linear gas velocity through the moving bed provides a much stronger correlation. Figure 4 illustrates the adsorption efficiency as a function of the linear gas velocity. The points represented in this graph are calculated based on the same 440 g/hr carbon repletion data used in Figure 3. It is apparent that minimizing linear gas velocity through the moving bed is an integral part of process efficiency.

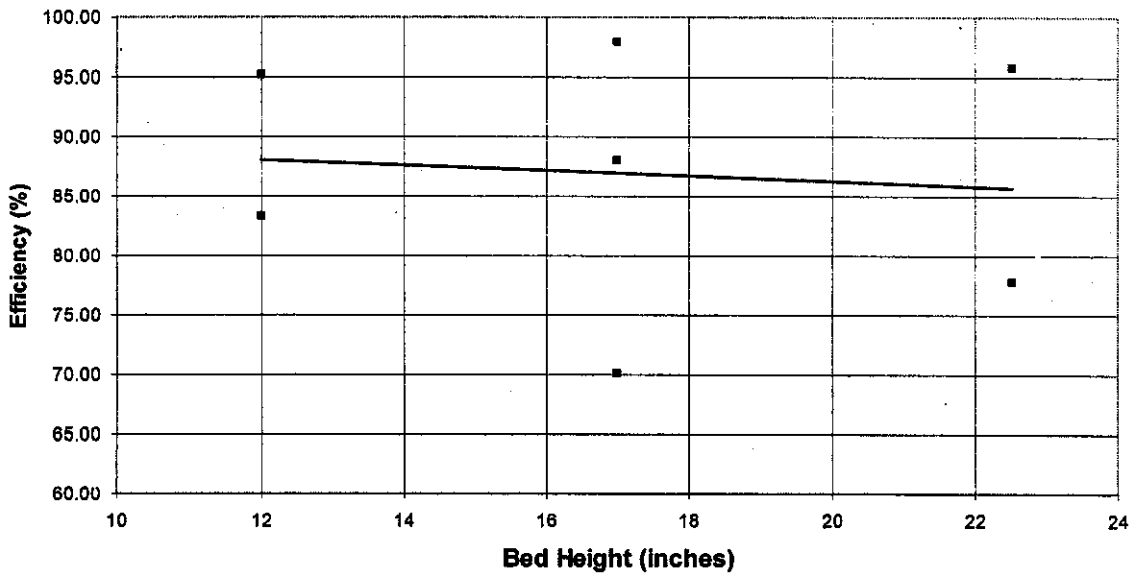


Figure 8: Adsorption Efficiency by Bed Height

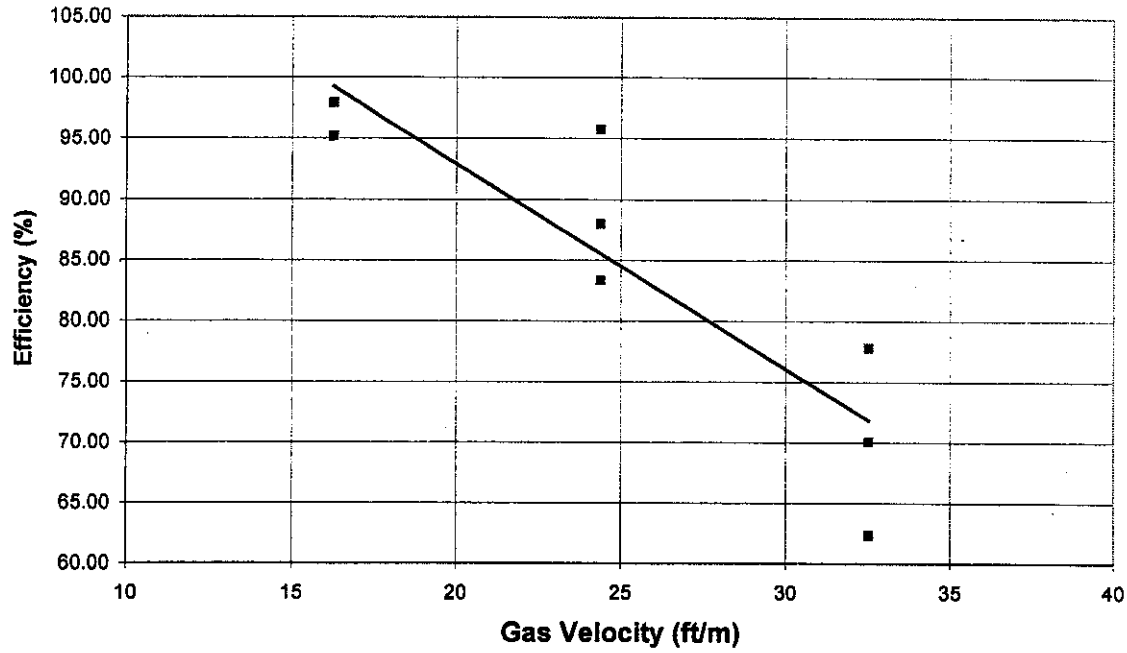


Figure 9: Adsorption Efficiency by Linear Gas Velocity through Adsorbent Column

Large Prototype Moving Bed Design and Construction

Based on the data obtained from the 5-inch moving bed testing, a larger prototype moving bed capable of handling 120 ACFM diesel exhaust flow is being designed and fabricated. The prototype moving bed will have an increased carbon adsorbent throughput consistent with the findings reported from the small moving bed testing. Bed volume will be largely diminished with respect to the original semi-batch prototype design.

Moving Bed Efficiency Correlation

To aid in the scale up operation for the 5-inch moving bed to the prototype moving bed operation, a preliminary correlation to determine adsorption efficiency as a function of dimensionless mass and residence time was prepared. The correlation

$$\eta \approx 100 \cdot \left[Exp\left(-\frac{0.8 \cdot \left(Exp\left(\frac{2}{t_r} \right) \right)^3}{m_{ratio}} \right) \right]^2$$

where

η = Adsorption Efficiency

t_r = Residence Time (seconds)

and

m_{ratio} = Dimensionless Mass Ratio [Adsorbent (g/hr) / Nox (g/hr)].

predicts adsorption behavior for the tested operating conditions with reasonable accuracy. For the tested samples, the correlation predicts performance within a mean average of three percent. Accuracy suffers under poor performance conditions (below 75% average performance). Improvements on the correlation for lower performance ranges are being investigated. The correlation is based on the boundary hypothesis that as the dimensionless mass ratio approaches infinity, the adsorption efficiency will approach 100%. As this ratio goes to zero, the efficiency will also go to zero. The second independent variable, residence time, is a function of linear velocity and bed height. It varies in a similar manner to the dimensionless mass.

Exhaust Gas Heat Transfer

Adsorption of NOx onto a carbon adsorbent requires bed temperatures below 300 °F. To improve adsorption efficiency, it is better if the temperature is further reduced below 250 °F. Diesel exhaust gas temperatures at the manifold for a fully loaded engine at steady state typically reach 1080 °F. Although the exhaust gas rapidly cools to approximately 500 °F after the first few feet of piping, mechanisms for cooling the exhaust gas to a temperature below the proscribed 250 °F will be necessary for the final prototype. A computer model simulating a finned heat exchanger was designed and utilized to help predict cooling efficiencies and potential difficulties in the exhaust cooling step.

Heat Transfer Model

The initial heat transfer system design plan was to force the heated exhaust gas through the annulus between the adsorption bed and a co-radial-finned cylinder surrounding the adsorber. By diminishing the annular space, the linear velocity of the exhaust flow would be increased, thereby increasing the heat transfer coefficient of the heat exchanger. With fins on the surface of the outer wall, ambient convection would remove the heat from the column and provide the impetus for further gas cooling. Figure 5 illustrates the co-radial design.

A simulation of this heat transfer design was programmed to test the feasibility of the concept. The simulation results revealed three potential concerns for creating a functional design. Foremost is the need for a very small annular space to provide sufficient linear velocity and gas-wall contact to obtain a heat transfer coefficient capable of cooling the gas. The required annular space is less than ¼ inch for a 15 inch OD outer shell. This may provide difficulties due to excessive pressure drop.

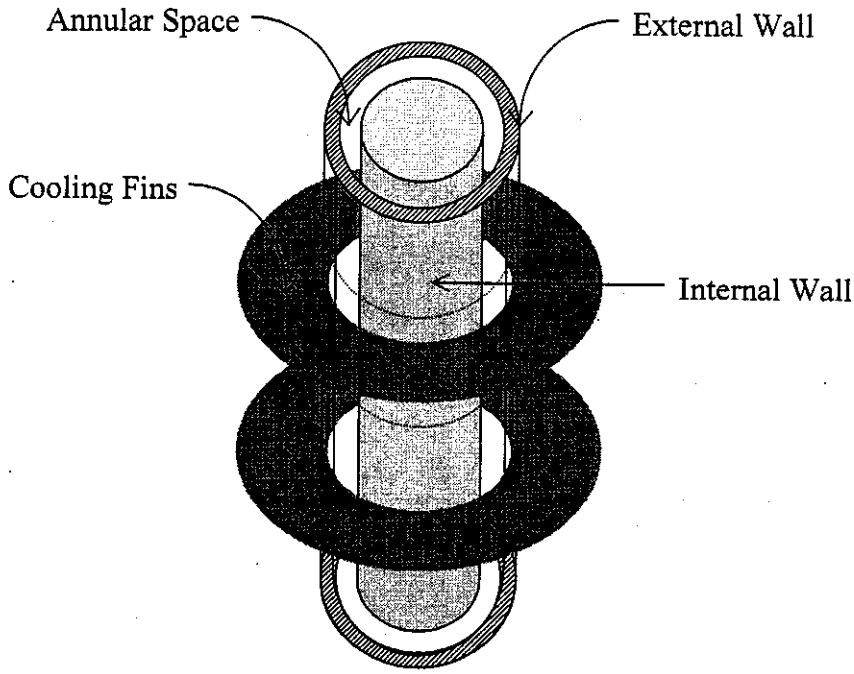


Figure 10: Co-Radial Heat Transfer Diagram with Annular Space

The second concern is that in the model, the interior co-radial cylinder acted as a heat sink to prevent gas cooling. As the hot exhaust gas entered the annular space, the gas heated the inner and outer walls at roughly the same rate. While the external surface was capable of shedding its heat through convection, the internal surface simply continued to heat up. This led to poor overall cooling performance, as the interior wall partially reheated the gas as it was being cooled through the external surface. To correct this problem, a layer of insulation could be applied to the inside cylinder. This would diminish the heat transfer to the interior cylinder and prevent the reheating effect. Such a scenario was modeled with improved results.

The final concern was the efficiency of the external cooling fins. The fins were not sufficient to cool the pipe under natural convection conditions. To achieve a heat transfer coefficient in the range necessary for successful gas cooling, a fan would have to be set up to rapidly blow air across the system, the system flow path length would have to be increased, or the length of the fins would have to be increased. Although each of these options is theoretically quite possible, from a logistical standpoint they are unfeasible. Adjusting the parameters in any of these ways to provide a satisfactory cooling design scheme would make the process design size prohibitive.

To overcome the cooling obstacles, a new cooling prototype design is being modeled and tested. In this model, the diesel exhaust gas stream is divided into 4 stream, each flowing through a 1-inch copper tube. The copper tubes meander through a radiator-style fin system coiled around the adsorption column. Although the fin efficiency is not greatly improved in this model, the flow path is increased ten fold without losing velocity or internal heat transfer. Pressure drop across the flow path would be lessened as well. Preliminary computer simulation results indicate that this design will perform favorably for the process conditions that will be present in the final prototype design.

Regeneration Apparatus Shakedown Testing

The regeneration apparatus applies microwaves to the NOx saturated carbon adsorbent to simultaneously regenerate the adsorbent and destroy the adsorbed NOx. Two regeneration devices have been employed in testing, the T-reactor and the meanderline. The T-reactor couples microwaves to a helix that surrounds a quartz reaction chamber. The carbon adsorbent is placed in the chamber where it is exposed to microwaves

and a nitrogen gas purge stream to prevent combustion. The meanderline system continuously deposits a thin layer of saturated adsorbent onto a conveyor belt that passes under a horizontal microwave field. A nitrogen purge is flowed through the microwave cavity.

Improved Moving Bed T-reactor

The initial prototype configuration employs a meanderline for regenerating the saturated char that is removed from the bottom of the contactor. After construction and testing of the meanderline apparatus, two practical design difficulties were realized. The primary concern with the meanderline was that it was difficult to achieve a sufficiently airtight conveyor path to fully prevent carbon combustion. As a result, carbon regeneration efficiency was decreased due to the adsorbent loss through combustion. Additionally, the combination of absorbed microwave power and combustion heat demanded high temperature conveyor belt materials for extended use. Although such materials are readily available, the associated costs are quite high.

As a result of the meanderline difficulties, a continuous regeneration T-reactor was designed to provide regenerated adsorbent at a rate sufficient for use with the prototype moving bed adsorption column. The T-reactor uses timer actuated lock hoppers to add and remove carbon adsorbent. As the carbon passes through the quartz tube it travels through the microwave field coupled to the helix surrounding the reaction bed. By changing the actuation period and energy input, adsorbent regeneration rate can be equated to adsorption column carbon repletion demands. This allows for the easy integration of the moving bed adsorption column with the regeneration apparatus to form one continuous cycle. The helical T-reactor regeneration device is presented in Figure 6.

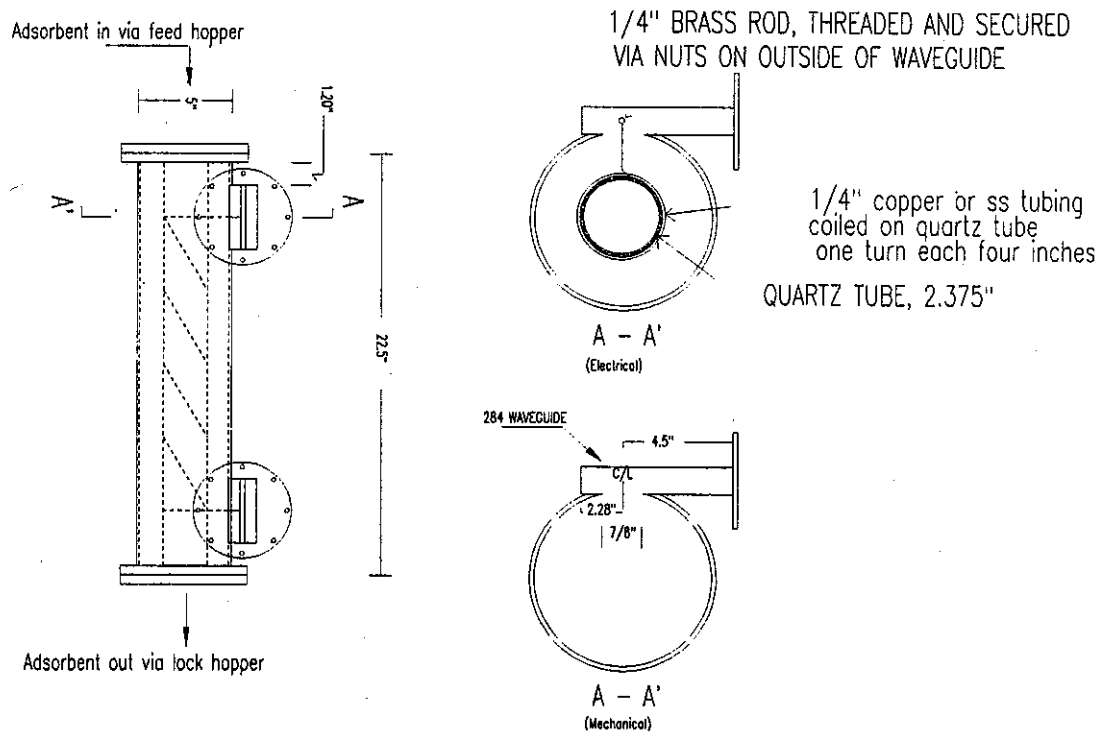


Figure 11: Helical Moving Bed T-Reactor

The continuous T-reactor design is more readily transported and streamlined than the meanderline system. This design improvement will further decrease the overall mass and size of the NOx destruction process.

Updated Process Flow Diagram

As a result of the improvements to the adsorption column and regeneration devices, the prototype process flow diagram has been updated. Figure 7 shows this new process design. The moving bed adsorption column and helical regeneration reactor are B-102 and B-103 respectively. Pumps G-101 and G-102 provide the vacuum necessary to transport the carbon adsorbent between the adsorption column and the regeneration reactor. Lock hoppers operated by timed actuation devices are situated at the top and bottom of both the adsorption column and regeneration reactor to control adsorbent transport. Feed hoppers F-101 through F-104 store the surplus adsorbent to maintain smooth actuation. Details of the major equipment used in the process flow diagram are provided in Table 3.

Identification	Size	Material of Construction	Source
B-101 Catalytic Converter	100 CFM	Stainless Steel Housing, Ceramic	Prototec Company
H-101 Soot Filter	5.625" dia. 6.0" long	Ceramic Monolith	NGK, Southfield, MI.
M-101 Microwave Generator	Variable 0 to 6000 Watts	Steel Cabinet	Cober Electronics, Inc. Model S6F
E-101 Air cooled heat exchanger	41,000 BTU/Hr	Galvanized Steel Aluminum Core	Xchanger, Inc. Model AA-250
B-102 Moving Bed Adsorber	100 CFM	Aluminum	CHA Corporation Custom Made
M-102 Microwave Generator	8" wide, 12' long, 6" high. 950 Watts	Steel Housing	CHA Corporation Custom Made
B-103 Microwave Regeneration Device	5-inch outside diameter, 30" high	Aluminum and Quartz	CHA Corporation Custom Made
M-103 Microwave Termination	1000 Watts	Aluminum	Cober Electronics
G-101 and G-102 Blower	Three inch Blower/Vacuum Pump	Steel with plastic housing	W.W. Grainger Model 6H004
F-101 through F-104 Adsorbent Storage Bin	100 pound capacity	Steel and Polypropylene	CHA Corporation Custom Made

Table 3: Process Flow Diagram Major Equipment List

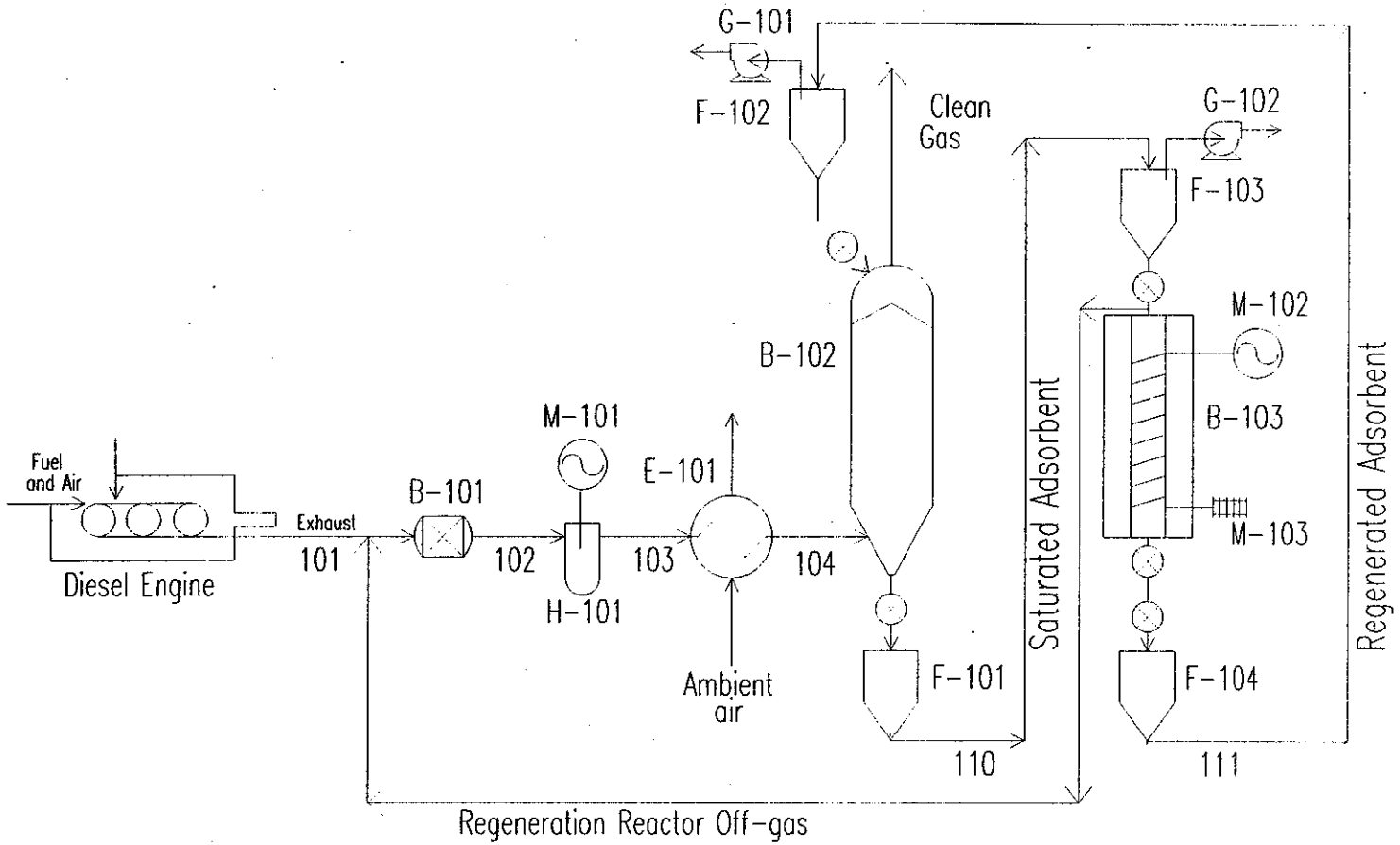


Figure 12: Updated Process Flow Diagram for Diesel Exhaust Pollution Control Device

Appendix E: Task 5: Soot Destruction Device Testing

Filter System Background

To be successful, a filter system should be durable, simple in construction and operation for reliability, cost competitive with other means of reducing particulate emissions, low-maintenance, and require little or no attention by the operator. Two filter types were used in experimentation, a ceramic monolith filter made by Corning, Inc and a particulate filter cartridge made with Nextel ceramic fibers by the 3M Company.

Nextel Filter System Background. The ceramic Nextel filter traps soot produced by diesel engine exhaust. Nextel fibers have a typical diameter between 10 and 12 microns and have a high elasticity and thermal capacity when compared to a ceramic monolith. The Nextel ceramic fibers are continuous metal oxide fibers made from alumina, silica, and boria. These filters are easily converted into ceramic textiles for high temperature operating environments. The 3M filters are available in three configurations. The basic filter cartridge has the Nextel fibers wrapped around a perforated metal support tube. Typically, the support structure is made of 18 gauge, 50% open perforated stainless steel. One end of the tube is plugged, forcing exhaust gases to flow radially through the Nextel media, thereby trapping the soot. The filter must then be externally regenerated. The 3M particulate filter cartridges can also be constructed by winding fibers around a metal electrically resistive heater. By closing one end of the heater, exhaust gases are forced through the filter media. The final design is available in a concentric tube pack configuration in which 3 or 5 filter cartridges are packaged inside progressively larger cartridges. This gives maximum surface area for a given volume. The melting point of the Nextel fibers is 1786 °C but regeneration temperature must be maintained at temperatures no higher than 886 °C to prevent damage to the fibers. The Nextel fibers have low thermal conductivity, thermal shock resistance, low porosity, and good chemical resistance. Ceramic fibers differ in properties from other commercially available inorganic fibers, such as fiberglass and silica. As a result of their high temperature properties, ceramic fibers are more suitable for diesel filter applications. These filters are resistant to thermal and mechanical shock due to the flexibility of the wound ceramic fiber construction. In general, several filter cartridges are assembled in a canister in order to make a complete diesel particulate filter system. The number and type of cartridge is dependent on exhaust flow rates, performance and regeneration intervals. Therefore, the design can be adapted to various engine sizes. In addition, the fiber that is within the filter can be reclaimed and manufactured into insulation materials.

Corning Ceramic Monolith Filter Background. The ceramic monolith filter made by Corning has a low thermal expansion, low pressure drop, and high efficiency. The filter material is the main difference between the 3M and Corning filters. The Corning filter is made from solid ceramic as opposed to fibrous ceramic. The Corning ceramics have a very low, near zero, thermal expansion coefficient, and can tolerate extreme thermal cycling. In addition, they have a high-temperature resistance, a high-temperature mechanical strength, and the yield strength actually increases as they are heated up to 1200 °C. As a result, the ceramic filters can retain their structural shape under stress at high-temperatures. The filters are inherently porous and can be easily equipped with washcoats and catalysts. Similar to the Nextel filter, the channels of the filter are plugged at one end and the exhaust gases are able to pass through the pores in the cell wall and exit the filter through an adjacent channel. The soot is then trapped on the connecting wall of the channels. After the filter is loaded with soot, it must be heated to oxidize the soot to carbon dioxide. After regeneration, the filter can be re-used.

Experimentation

To test the two different styled soot filters, several experiments were conducted that established the ability of the filters to capture soot and then be regenerated with microwaves. For CHA experimentation, the ceramic monolith filter was impregnated with an oxidation catalyst, and a 3M filter made of Nextel fibers was prepared. Both filters were wrapped in a microwave helix configuration to accommodate microwave application. Figure 1 illustrates the two filters used in this series of experiments.

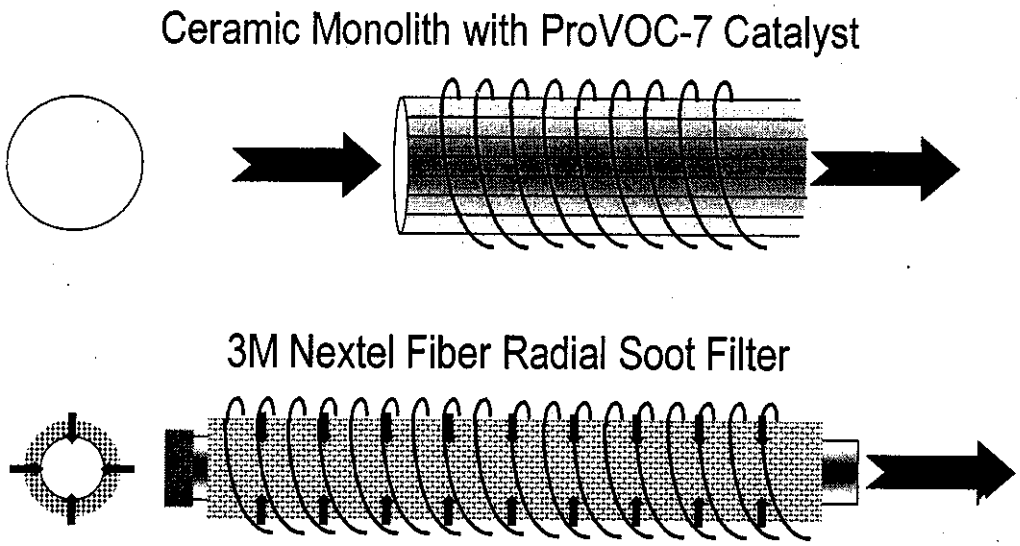


Figure 13: Diesel Exhaust Soot Filters

A 58 hp diesel test engine was used to source exhaust gases. During each run, exhaust gas was flowed through either the 2-inch 3M filter or the ceramic monolith filter at a rate of 70 CFM. The temperature was held constant at approximately 300°F. As more soot collected onto the filter, the pressure drop across the filter increased. During flow, the pressure drop across the filter was monitored to establish filter saturation. Figure 2 depicts this pressure drop versus time for each of the filters as well as for multiple regenerations.

A variety of filter housing and microwave coupling configurations were tested and the better configuration selected for use in the prototype. Microwave energy was applied to the monolith filter during both flow and after the flow period in an attempt to achieve continuous saturation/ regeneration in the soot filters. The maximum pressure drop was 45 inches of H_2O.

Filter Regeneration

After the filters were saturated with soot, they were exposed to between 750 and 900 Watts of microwave energy with an air flow rate of 4.5 SCFH. Both results were achieved by regenerating the filter off line with an air flow to support soot oxidation. The microwaves excite the small soot particles and create interparticular friction. This friction translates the microwave energy into heat energy providing the impetus for soot oxidation. As a result of the oxidation mechanism, sufficient soot accumulation is a requirement for successful regeneration.

Results and Discussion

Experimentation has shown that both the 2-inch 3M filter and the ceramic monolith filter can be regenerated using microwave energy. The ceramic monolith filter has a collection efficiency of approximately 85%, while the 3M filter has a collection efficiency of >90%. Although the 3M filter has a higher collection efficiency, it is not as easily regenerated as the ceramic monolith filter. The best microwave configuration for the 3M filter was directly wrapping the filter body with a copper helix. The ceramic monolith, wrapped directly with a helix, was easily regenerated. Eeach regeneration did not degrade the filter's ability to capture soot, as is apparent in Figure 2. However, the ceramic monolith that was not coated with an oxidation catalyst was not easily regenerated. In fact, it was regenerated less effectively than the 3M Nextel filter.

Unfortunately, the ceramic monolith was accidentally broken before extensive experimentation could be completed. Three more ceramic monoliths were purchased for further testing. From the initial testing, it was apparent that the ceramic monolith filter was better suited for the needs of the device under development

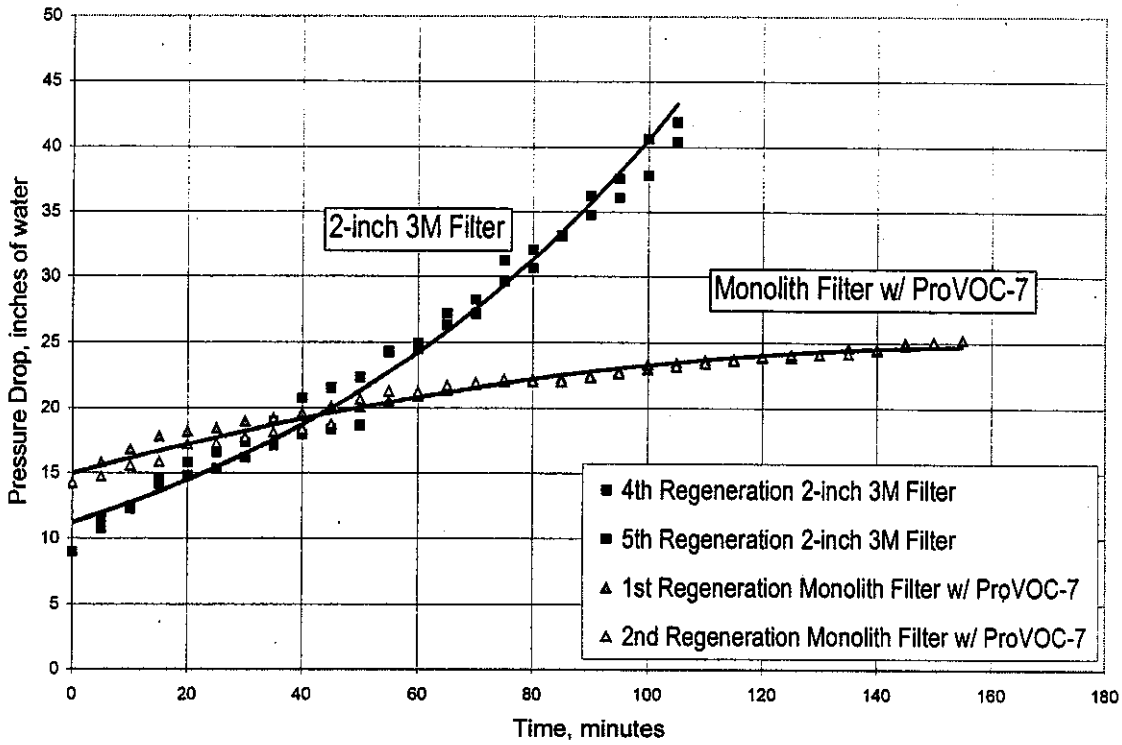


Figure 14: Soot Filter Pressure Drop and Regeneration Comparison

Figure 3 shows a schematic of the dual magnetron monolith design. This configuration has shown the best results so far. This device is six inches long and 5 5/8 inches in diameter and centered in the cavity with three inches of open space on either end. The two inch magnetrons are placed directly over the ends of the cavity. The diesel exhaust enters the cavity and is passed through the filter end to end.

Five saturation/ regeneration cycles have been completed so far using the ceramic monolith filter with 70 SCFM of diesel exhaust. During regeneration, the filter is off line and a constant stream of 4.4 SCFM air purge was passed through the filter. The experimental conditions for each test is outlined in Table 1. For regenerations 2-5, the magnetrons were left on and microwave energy was introduced during saturation. Asynchronous microwave application represents alternately operated magnetrons. The temperature status refers to the temperature of the filter at the start of regeneration. During a "hot" temperature state, regeneration took place immediately following saturation and during a "cold" temperature state, regeneration did not occur until the system had cooled after saturation was completed. Saturation was considered complete when the manifold pressure exceeded 45 inches H_2O while regeneration was considered complete once the outlet oxygen concentration was equal to the inlet oxygen concentration.

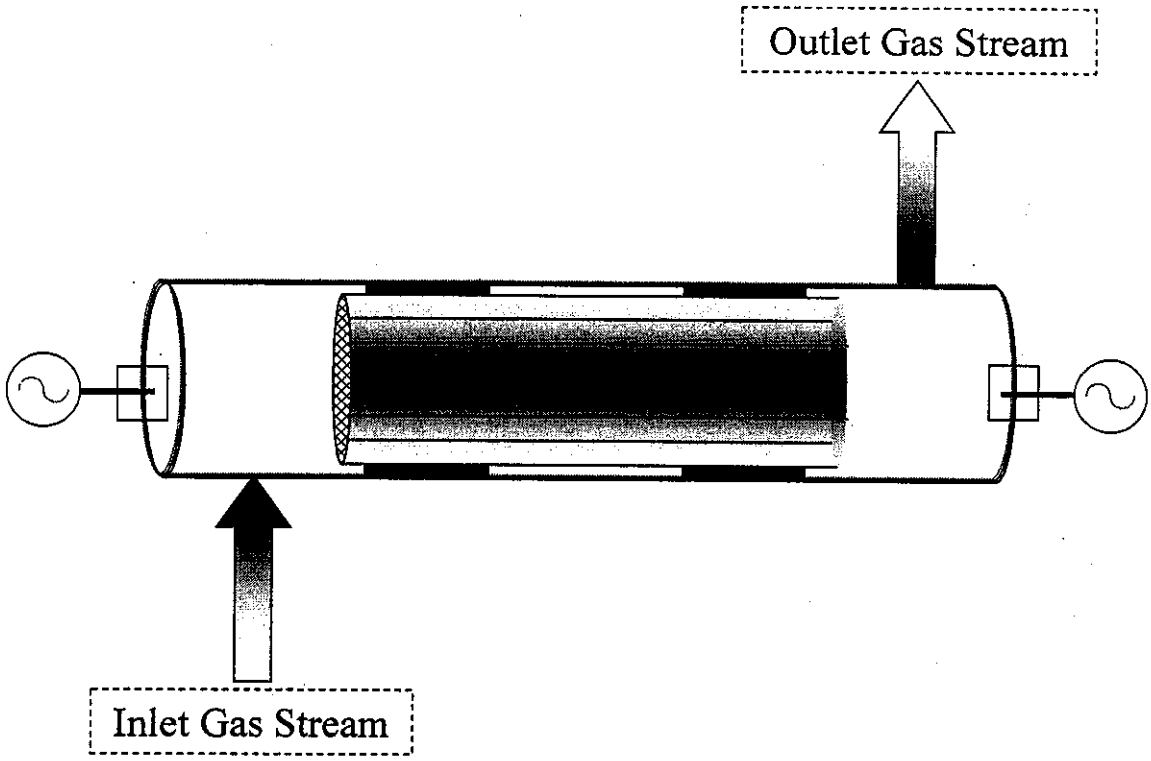


Figure 15: Ceramic Monolith Filter with Cavity Microwave Application

Table 4: Monolith Soot Filter Regeneration Test Parameters

Cycle	Saturation			Regeneration	
	Time (Minutes)	Microwave Application	Temperature Status	Time Minutes	Carbon Consumed Grams
1	46	Off	Hot	95	1.806
2	55	Continuous	Cold	95	3.525
3	125	Continuous	Hot	60	5.139
4	88	Continuous	Hot	100	2.582
5	200	Continuous	Hot	60	5.464
6	360	Asynchronous	Hot	50	4.210

Figure 4 depicts the 3rd, 5th, and 6th regenerations of the ceramic monolith filter using the configuration shown in Figure 3. During each of these three regenerations, saturation was completed with microwave power applied. Additionally, these runs were saturated for an extended time as shown in Table 1. The shape of the curve shows that once a maximum accumulation of soot is in the filter, the microwaves successfully promote oxidation. An equilibrium is reached where the amount of soot available equals the amount of soot necessary to absorb microwave energy for combustion. The filter can then be continually loaded with a constant manifold back pressure. The 3rd and 5th regeneration reached a similar soot density, 5.14 grams and 5.46 grams respectively, at which point the soot was able to begin combustion, as the manifold back pressure reached 45 inches of water. The soot density before combustion begins in the 6th regeneration is slightly lower at 4.21 grams. For the 6th regeneration, a nozzle was placed over the magnetron to further facilitate the penetration of the microwaves. Due to this increased penetration, the excess soot was able to combust at a lower manifold back pressure and the filter saturated for a significantly longer period than previous regeneration/ saturation cycles. In fact the 6th regeneration sample never achieved a manifold back pressure of 45 inches of H_2O, having reached a steady state at approximately 41 inches, this explains the lower carbon consumption quantity during regeneration, as the filter never actually saturated.

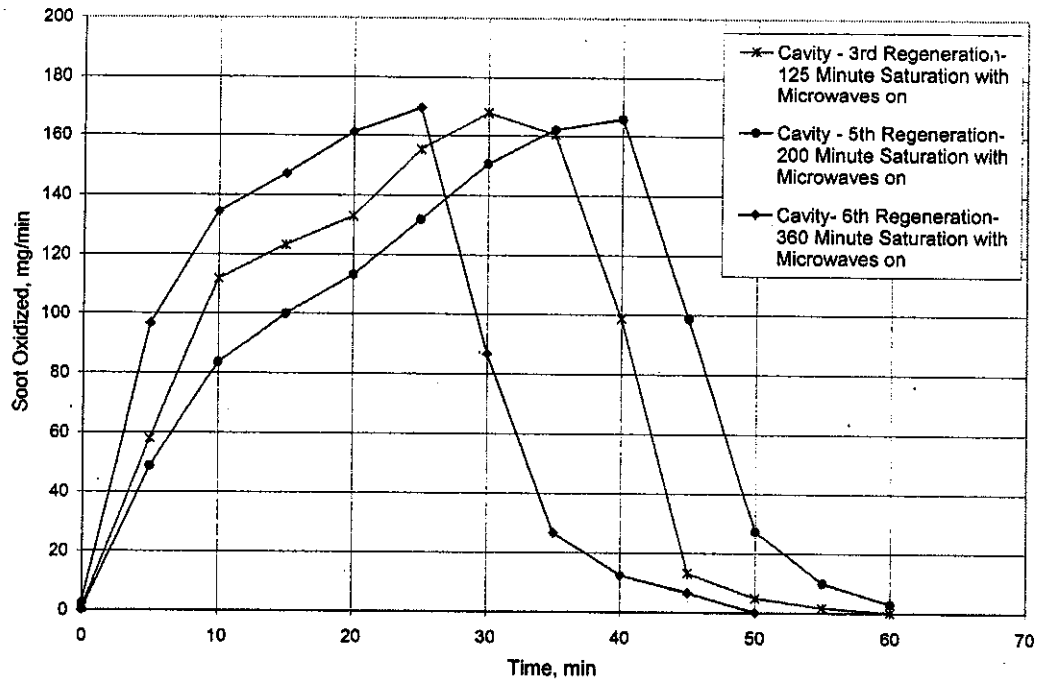


Figure 16: Monolith Soot Filter Regeneration for Extended Saturations with

Continuous Microwave Application

Figure 5 illustrates the effect of microwave power during saturation on regeneration rate. The 1st regeneration was completed after the filter had been saturated without continuous microwave power. Saturation was completed after 46 minutes without continuous microwave application. The amount of soot on the filter after the first regeneration was significantly less than the amount of soot on the filter after the 3rd regeneration. Interestingly, the 3rd regeneration was completed after the filter had been saturated with continuous microwave power. The amount of carbon that was loaded onto the filter after the third regeneration is two and a half times greater than the amount of carbon that was loaded onto the filter after the first regeneration. This corroborates the previous analysis indicating the need for sufficient soot accumulation for effective regeneration.

It is important to note that when the microwaves are applied throughout the saturation period, the amount of soot present on the filter after saturation times of 125, 200, and 360 minutes were nearly identical. The lowest amount of carbon consumed is represented in 360 minute saturation test. This evidence indicates that extended filter loading is beneficial for enhancing regeneration time and suggests the possibility of continuous loading and microwave regeneration without the need for off-line regenerating of the soot filter.

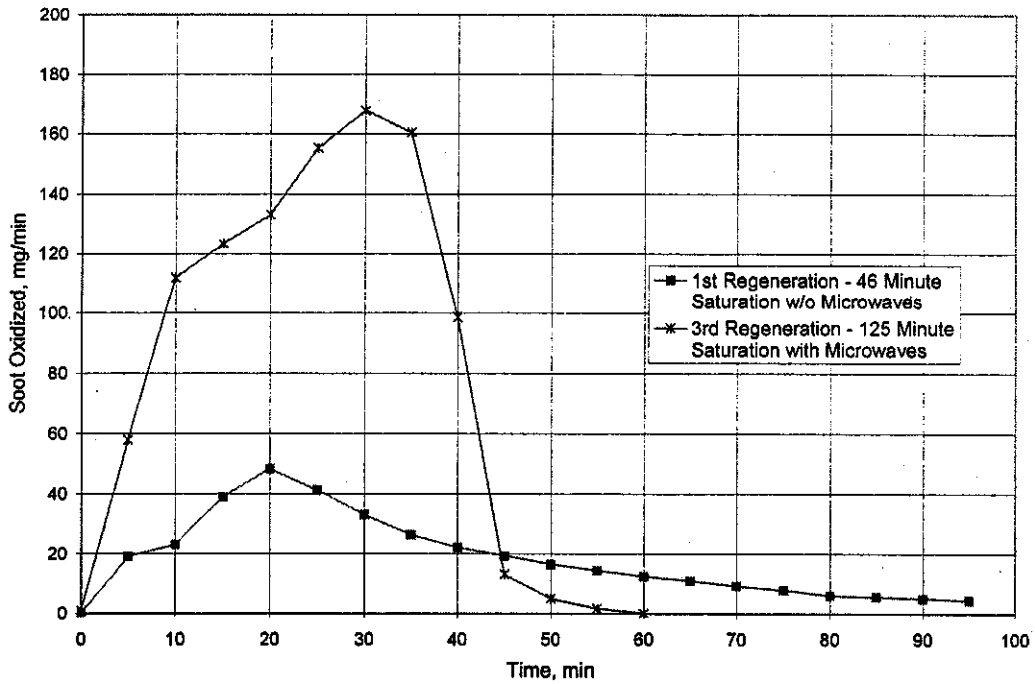


Figure 17: Monolith Soot Filter Regeneration for Extended Saturation

Figure 6 portrays the difference between hot and cold regenerations. The two curves shown in Figure 6 show a significant contrast between the cold start and hot start regeneration. In a cold start regeneration, the filter is allowed to cool after saturation, whereas a hot regeneration takes place immediately after the filter has been saturated. It is better to have the filter hot during regeneration because rapid oxidation does not occur with a cold regeneration. With a hot filter, it is much easier to reach the activation energy necessary for soot to combust. With a cold start regeneration, the energy necessary to reach the activation energy is much greater because the soot in the filter is not energized at all.

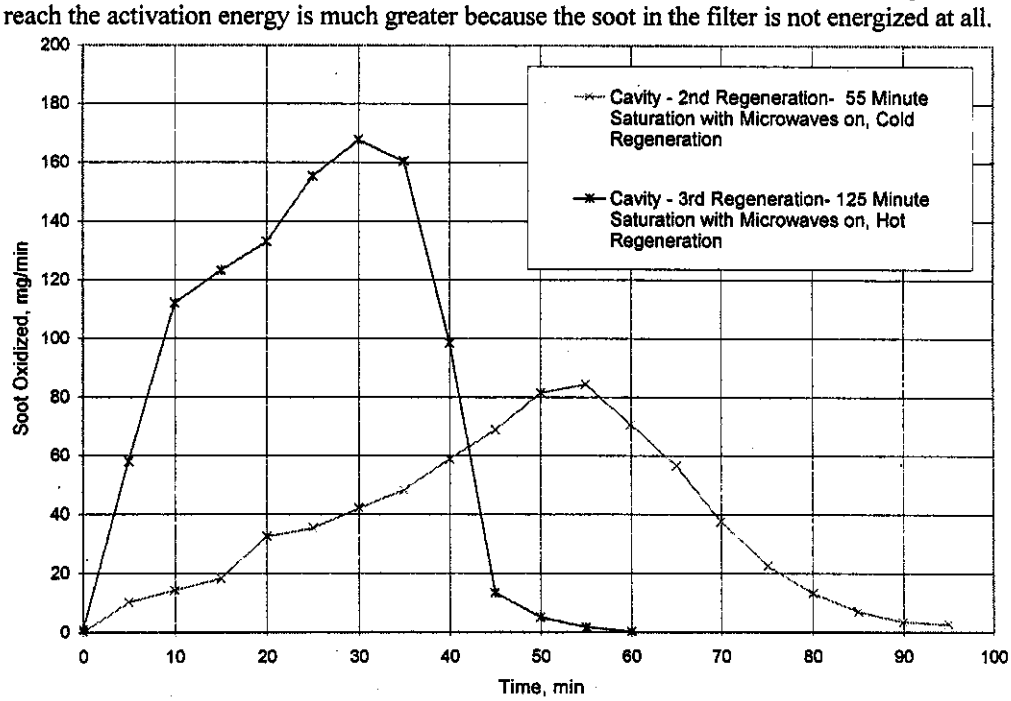


Figure 18: Hot and Cold Start Monolith Soot Filter Regeneration

Figure 7 illustrates the ceramic soot filter pressure drop as a function of time during six saturation cycles. Details of these saturation cycles can be referenced in Table 1. During subsequent saturations, the time required to 45 inches water back pressure increases. Figure 8 isolates the third, fifth, and sixth saturations to more clearly illustrate this trend. These saturation experiments strongly suggest the propensity for continuous soot destruction without need for off-line regeneration. As indicated by the regeneration experiments, heavy filter loading corresponds to increased soot microwave adsorption efficiency thereby facilitating rapid soot oxidation. As the soot accumulates, the system appears to approach equilibrium between incoming soot and the amount of soot necessary to provide adequate absorption of the continually applied microwave energy to facilitate combustion of some soot. After this equilibrium is reached, the filter can be used. This condition is favorable to eliminating the need for multiple filters and off line regeneration, if a system with sufficiently low equilibrium pressure drop can be designed.

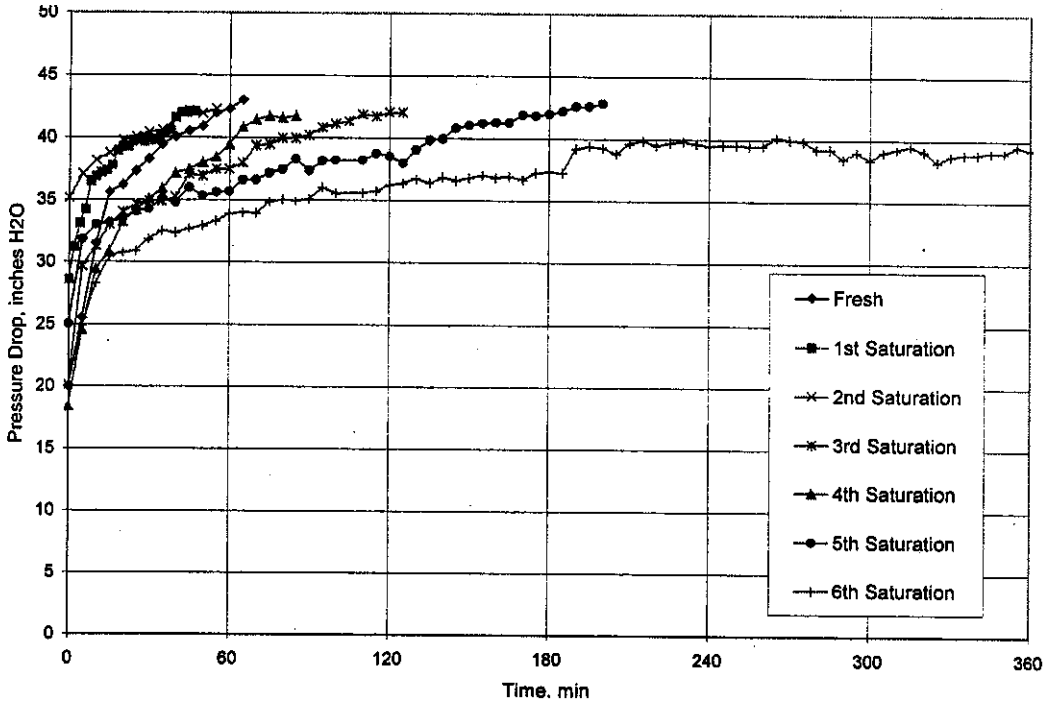


Figure 19: Monolith Soot Filter Pressure Drop by Regeneration

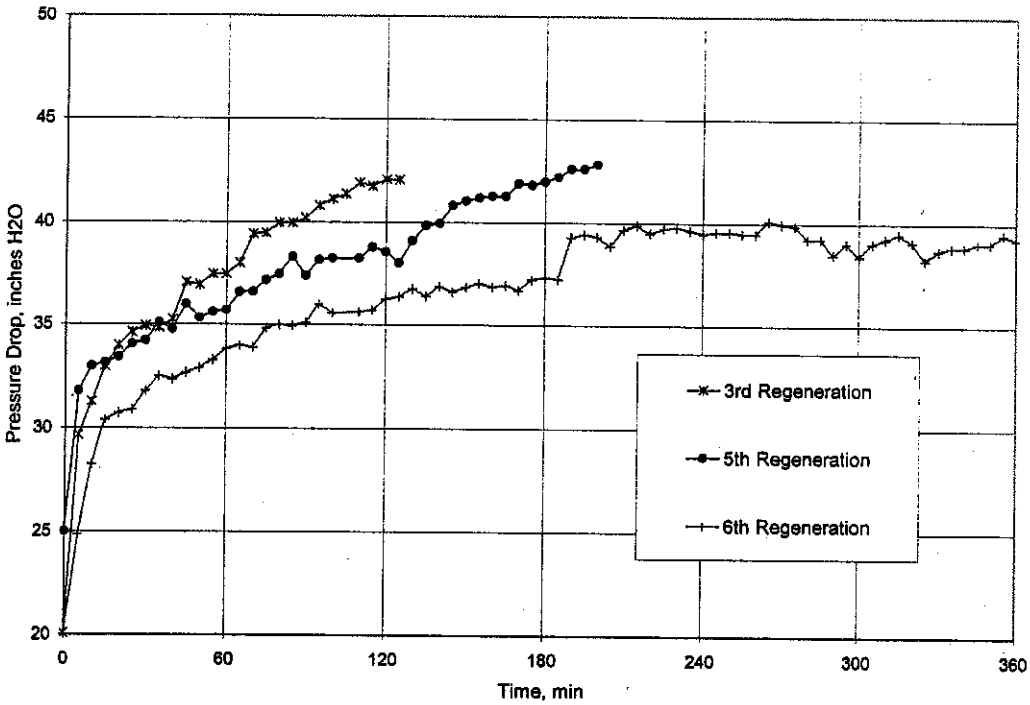


Figure 20: Pressure Drop for Extended Saturation with Monolith Soot Filter

Integrated CHA Diesel Exhaust Treatment Prototype Demonstration System

The integrated systems shown by the process flow diagram in Figure 9 has been assembled and is currently ready for testing. The integrated systems represent a continuous process of adsorption and regeneration through which diesel exhaust gas can be continuously cleaned. The saturated carbon adsorbent is passed through a regeneration column where microwaves are used to regenerate the carbon. Diesel exhaust flows into the system at 500 degrees Fahrenheit and 120 CFM. As is apparent in the schematic, diesel exhaust gas first encounters an oxidation catalyst bed that destroys and oxidizes 100 percent of the VOCs and CO apparent in the exhaust gas. The gas then flows into a microwave regenerated soot filter where more than 90 percent of the soot is removed. The oxidation and soot filtration stages may eventually be integrated. After the gas passes through, the filter can be completely regenerated. The gas is then routed through a heat exchanger for cooling. The gas is cooled from a temperature of 500 degrees Fahrenheit to a temperature of between 150-200 degrees Fahrenheit to better facilitate the adsorption of NOx onto the carbon adsorbent. The gas enters into the bed of carbon adsorbent where the NOx is adsorbed onto the carbon. The clean gas then exits through the top of the adsorbent bed. Before the NOx laden adsorbent is passed into the regeneration column, it is purged with a nitrogen stream to prevent adsorbent oxidation. The adsorbent passes into a hopper and is then introduced into the 60 mm ID regeneration column. Once in the regeneration column, the NOx laden carbon is exposed to microwaves under a nitrogen purge to desorb the NOx and regenerate the carbon adsorbent. The concentrated NOx stream then flows into a reduction catalyst bed where the NOx is destroyed. The regenerated adsorbent is then passed into a $1ft^3$ hopper. The bulk density of the carbon adsorbent is 30.5 lbs/ft^3. Therefore, in each cycle, the hopper can hold 30.5 lbs of carbon. The regenerated carbon cycles back into the adsorbent bed where it awaits the next round of NOx destruction. Testing is now being performed to determine the time interval necessary to completely regenerate the carbon adsorbent. Experimentation is currently being conducted with activated carbon and char to delineate which adsorbent is better suited for CHA and ICAT needs.

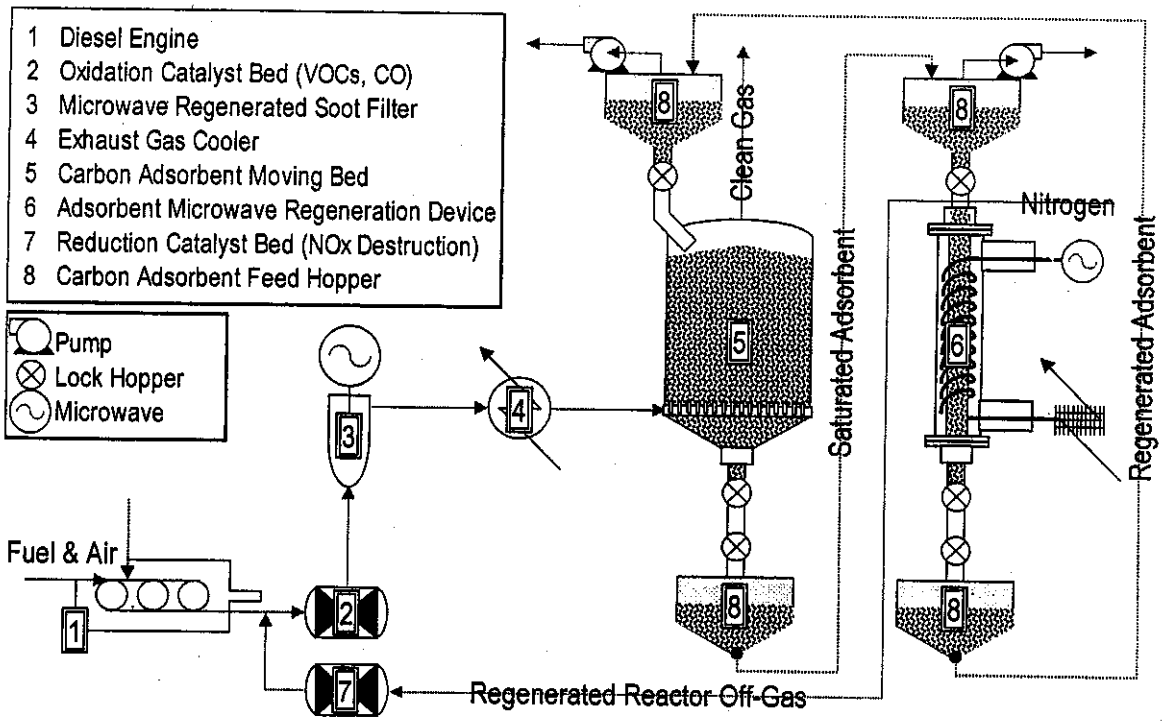


Figure 21: CHA Diesel Exhaust Treatment Process Flow Diagram for Prototype Demonstration

The updated integrated prototype design shown in Figure 9 is based upon suggestions by McClellan Air Force Base personnel to produce a continuously regenerated moving bed system that would allow for uninterrupted operation. The various components are depicted and described on the following pages.

Experimentation

Currently, the major components of the adsorption/regeneration process are assembled and the unit is ready to be tested. The previous system was based upon the batch adsorption and regeneration. After preliminary testing and consultation with the McClellan Air Force Base personnel, it was determined Filter testing is being conducted to determine the appropriate filter type and the regeneration capability.

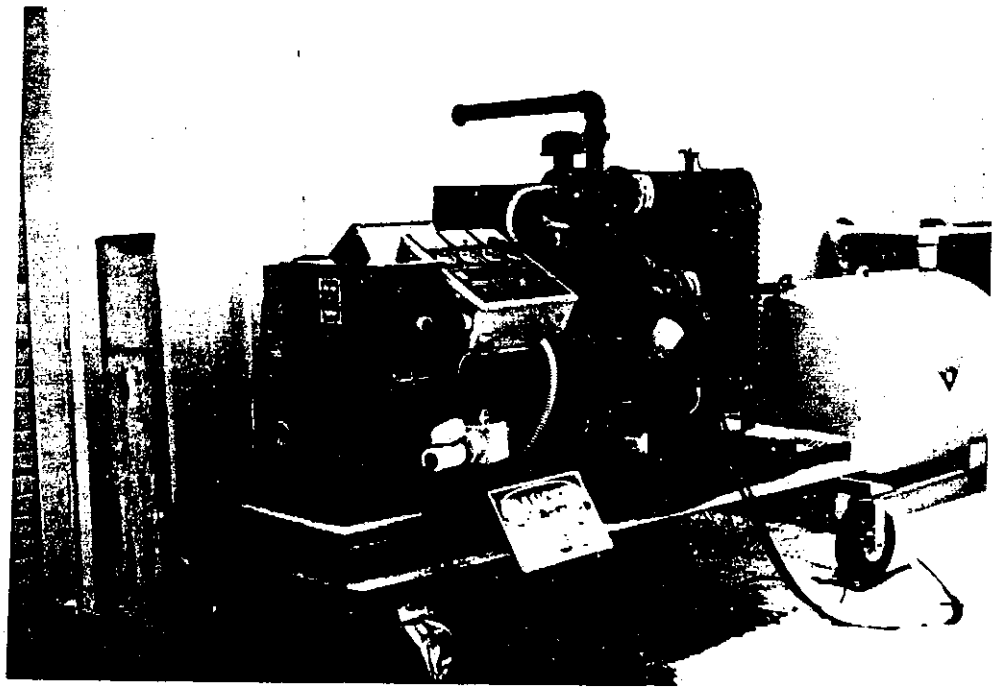
Experimentation was conducted to determine the minimum amount of time necessary for the carbon adsorbent to be in the presence of microwaves for full regeneration. Saturation was completed with a 22.5 inch bed of 10-mesh FMC char and a diesel exhaust flow of 2.5 CFM. A ten minute actuation time was necessary to achieve a 90% NO adsorption rate with the FMC char. Regeneration with microwaves was completed in a 60mm ID quartz tube with 1 SCFH flowing into the bottom of the tube and then combining with a nitrogen purge for a total flow of 2 SCFH out the top of the tube. The regenerated char was saturated once again in a bench scale adsorption column to determine the amount of NO the regenerated char would be able to adsorb. A tube 12 inches long with a one inch ID was filled with the regenerated char and a NO stream was combined with compressed air and flowed through the char at flow rates between 2.4-3.1 SLPM.

Results and Discussion

As indicated in Figure 10, the 750 Watt and 500 Watt lines are representative of char that has not been regenerated. The data shows that there was little saturation due to a low regeneration rate at these two power levels. The only two test conditions that show positive adsorption capacity gains are the 15 and 20 lbs/ hr regenerations at 1000 Watts. This indicates that there is a minimum necessary desorption energy for the char, as expected. Testing of higher power levels and duplicate testing for GAC samples is forthcoming.

1. Diesel Engine

The diesel engine pictured is a 58 hp, naturally aspirated diesel test engine. It operates at 1800 rpms and generates approximately 120 CFM exhaust gas. It is coupled to a 3 phase electric generator to which a load can be applied through a resistive heat bed. Exhaust gas is sent through the wall of the laboratory to where the integrated prototype system is assembled.

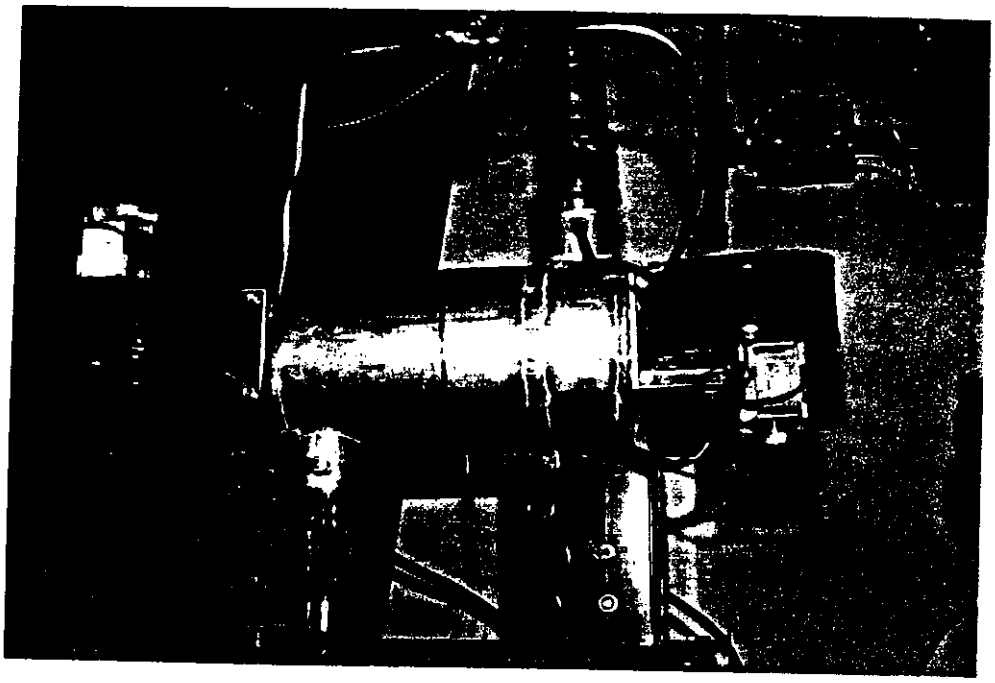


2. Oxidation Catalyst Bed

The oxidation catalyst bed pictured is attached directly to the diesel engine manifold during prototype testing. It contains a ceramic monolith coated with an oxidation catalyst. Testing has shown that the catalyst effectively destroys over 90% of the CO present in the engine exhaust. This catalyst device is also effective at reducing effluent VOCs.

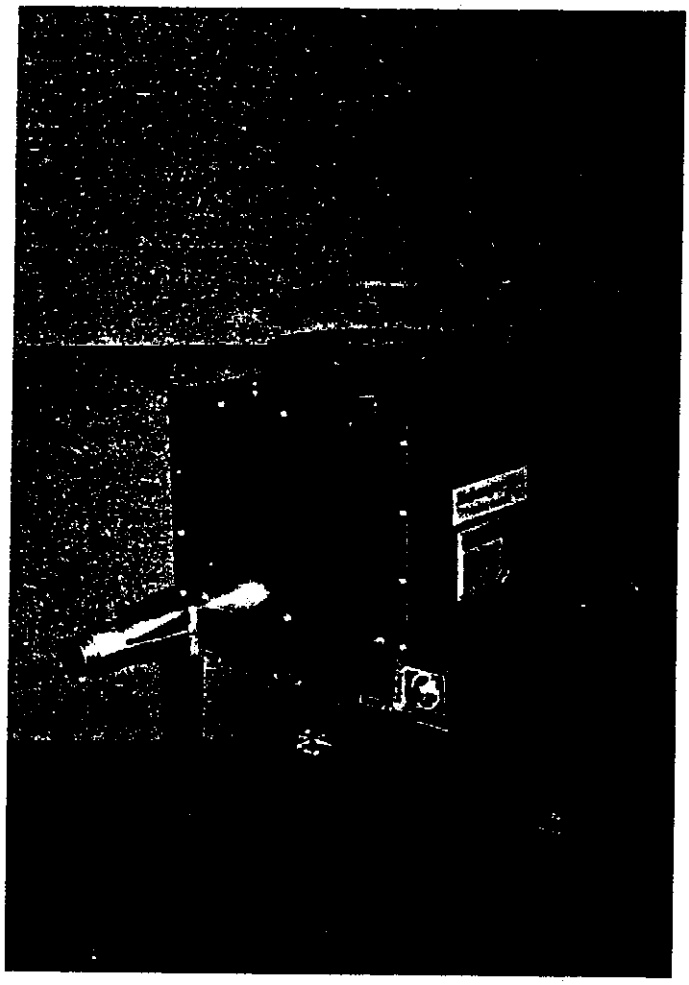
3. Microwave Regenerated Soot Filter

The microwave soot filter apparatus depicted is of the design used for testing the monolith soot destruction filters discussed in the previous section. The apparatus houses a ceramic monolith coated with an oxidation catalyst. Magnetrons at either end of the device introduce the microwaves into the monolith cavity. The 2.45 GHz magnetrons typically generate 800 Watts of microwave power. Cooling fans are attached to each magnetron to prevent overheating due to reflected microwave power.



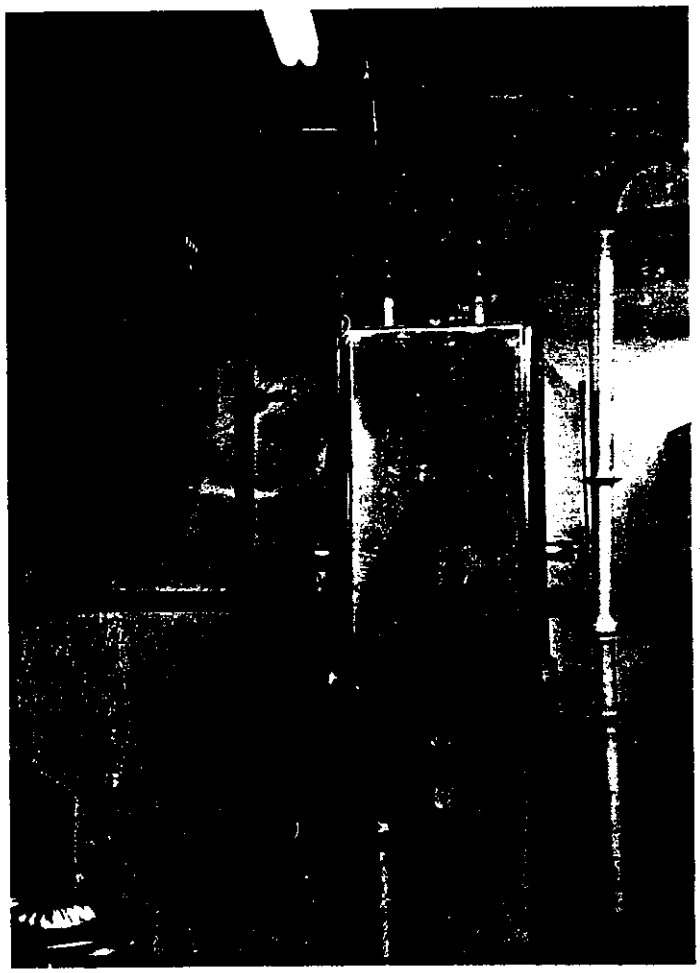
4. Exhaust Gas Cooler

The exhaust gas cooler below was designed to cool 120 CFM of gas from 500 °F to 100 °F. The heat exchanger is equipped with a variable speed electric motor to control cooling capacity of the heat exchanger. The exchanger is outfitted with 2 inch NPT inlet and outlet gas fittings. The unit is made of aluminum and is approximately 2 feet by 2 feet and stands 3 feet tall.



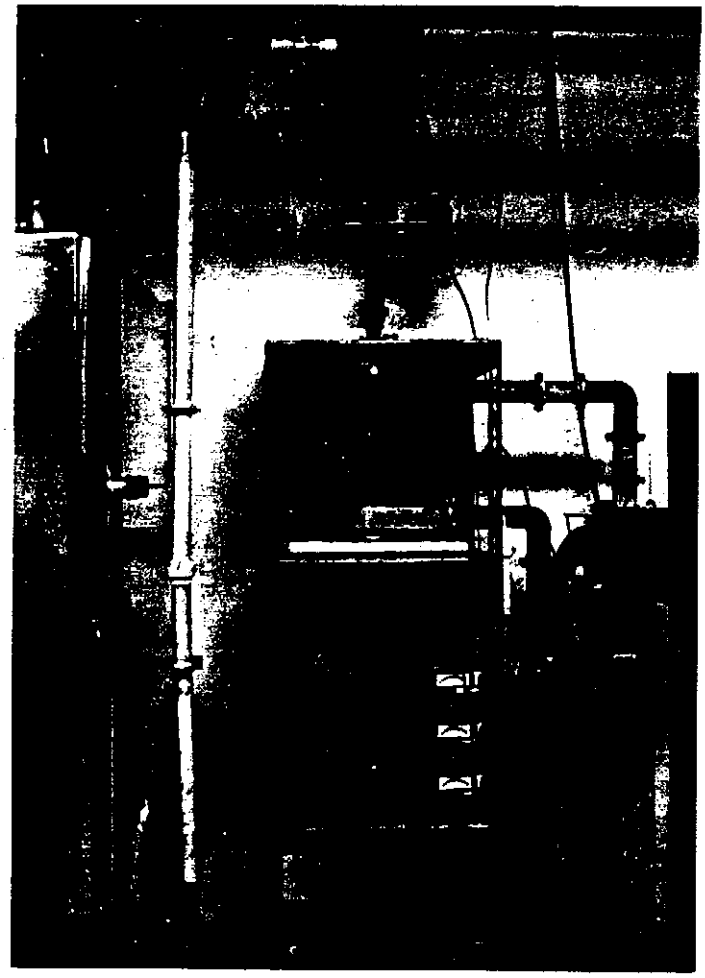
5. Carbon Adsorbent Moving Bed

The carbon adsorbent moving bed is 30 inches square and 40 inches tall. The carbon bed within the adsorption unit is 29 inches tall. The gas is introduced through two 2 inch NPT inlet manifolds. The carbon is continuously fed through the adsorption unit via a feed hopper at the top and exits through a lock hopper at the base.



6. Adsorbent Microwave Regeneration Device

The adsorbent regeneration device has a 5 inch outer diameter and is 22 inches tall. The helix equipped cylinder houses a 2 3/8 inch ID quartz tube which is extended the full length of the interior of the reactor. Carbon is fed by a feed hopper at the top and exits by lock hopper actuation at the base. The carbon is transferred between the two systems by means of two pneumatic conveyors, which are periodically operated to transfer the carbon when the receiving hopper is full.



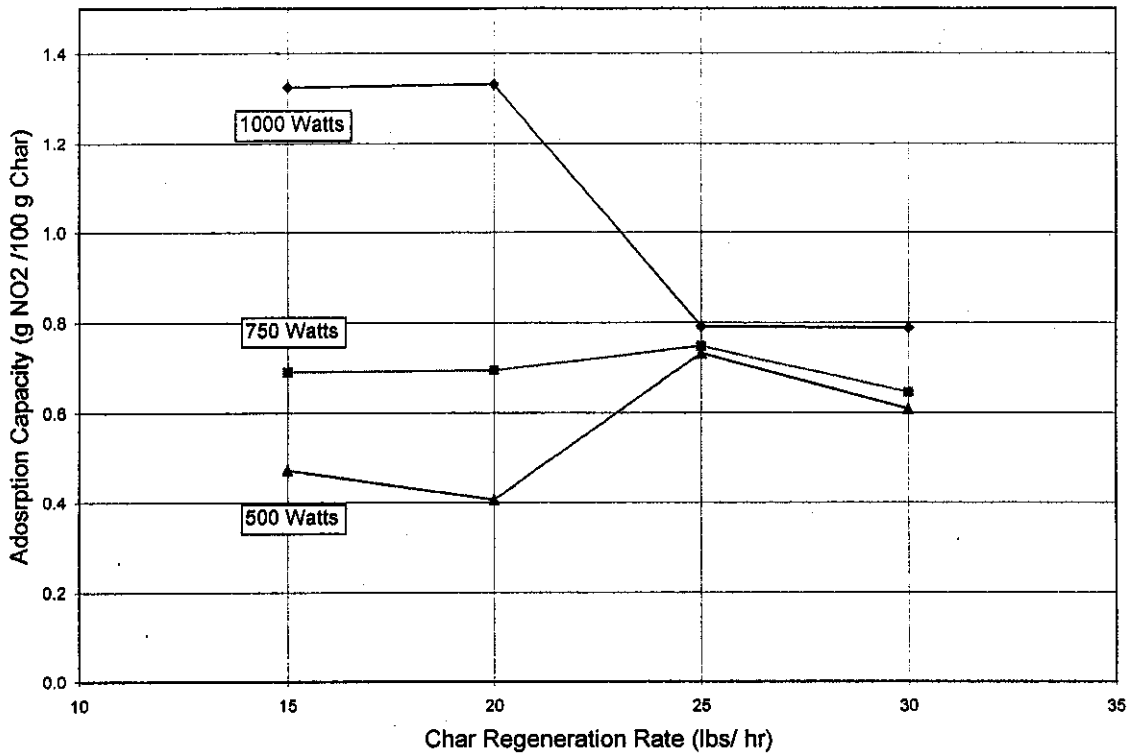


Figure 22: Microwave Regeneration Efficiency by Power and Char Throughput

Appendix F: Task 6: Fabricate Regeneration Device and Build Heat Exchanger

Integrated CHA Diesel Exhaust Treatment Process

The process flow diagram in Figure 1 shows the overall schematic of the CHA diesel exhaust treatment process system. The CHA diesel exhaust treatment process is a continuous process of adsorption and regeneration. The saturated carbon adsorbent is passed through a microwave field where the adsorbed pollutants are destroyed. Figure 1 shows the current process flow diagram for the prototype NOx treatment system. Diesel exhaust flows into the system at approximately 500 degrees Fahrenheit and 120 CFM. This hot gas passes through an oxidation catalyst bed (2) where VOCs and CO are converted to CO_2 and H_2O. The gas then flows into a microwave regenerated soot filter (3) where more than 90 percent of the diesel soot is removed. The gas is then passed through a heat exchanger (4) where the gas is cooled from 500 degrees Fahrenheit to between 150-200 degrees Fahrenheit to better facilitate the adsorption of NOx by the carbon adsorbent. The cooled gas enters the bed of carbon adsorbent (5) where the NOx is removed. Clean gas exits through the top of the adsorbent bed. Spent adsorbent continuously passes into a storage hopper located at the base of the adsorbent bed and is periodically transferred to the regeneration feed hopper (8. After passing through the microwave reactor (6) the regenerated carbon is stored in a one cubic foot hopper. The density of the carbon is 30.5 pounds/cubic foot. Therefore, in each cycle, the hopper can hold 30.5 lbs of carbon. The regenerated carbon cycles back into the adsorbent feed hopper where it awaits the next round of NOx adsorption.

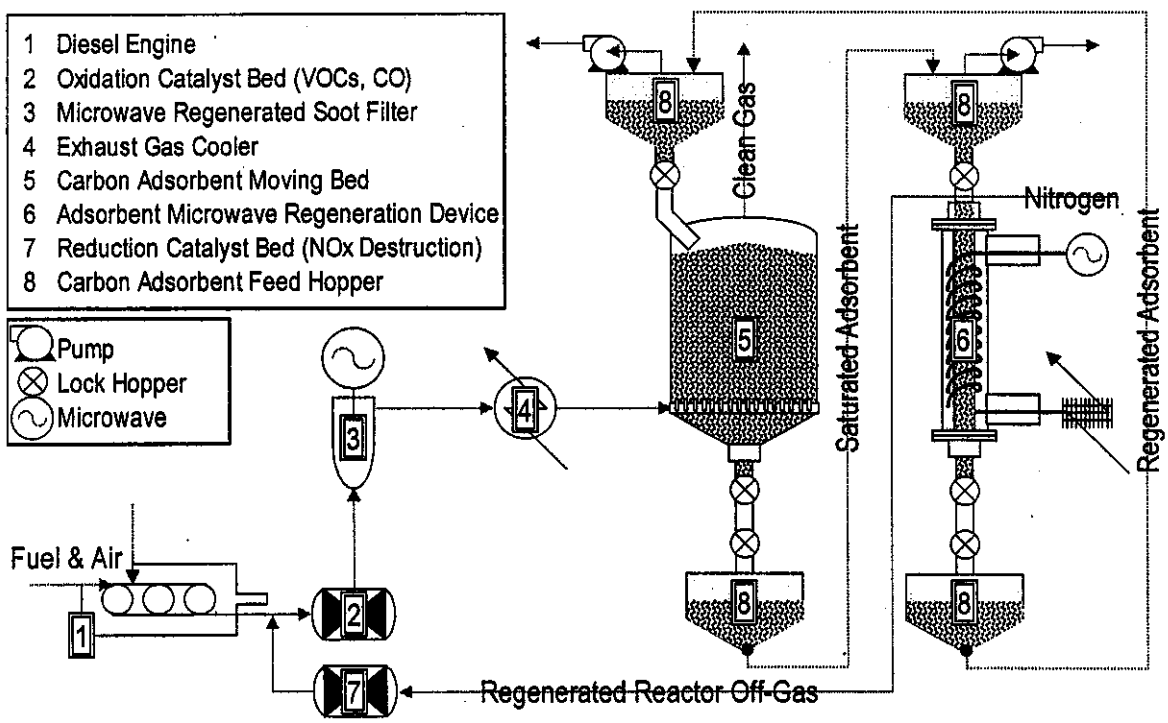


Figure 23: CHA Diesel Exhaust Treatment Process Flow Diagram for Prototype Demonstration

The fabrication of the adsorbent microwave regeneration device and the installation of the heat exchanger have been completed. The system is now ready for shakedown testing.

Adsorbent Microwave Regeneration Device

The adsorbent regeneration device is shown in the photograph below. The adsorbent regeneration device has a 5 inch outer diameter and is 22 inches tall. The helix equipped cylinder houses a 2 3/8 inch ID quartz tube which is extended the full length of the interior of the reactor. A feed hopper at the bottom of the reactor feeds carbon by lock hopper actuation. The carbon is transferred between the two systems by pneumatic conveyors, which are periodically operated to transfer the carbon when the receiving hopper is full.

The 2.3-inch quartz reactor equipped with helix has been constructed for microwave induced regeneration of NOx saturated carbon. This reactor is installed and ready for service. At the same time, we have constructed a second carbon reactor to be used for testing separate from the integrated system. Originally, it had been our intention to utilize char as an NOx adsorbent. However, initial economic evaluation indicates that the char will be the main operating cost because the NOx reacts with the char and continuously consumes a portion of the char during regeneration. On the other hand, we discovered that activated carbon will desorb the NOx, as opposed to reacting, because of the larger pore size of activated carbon. Furthermore, the activated carbon has almost twice the NOx adsorption capacity. We decided to experiment with the activated carbon to determine how suitable it would be for the CHA diesel exhaust treatment process. Adsorption studies with activated carbon have been completed using 5-inch moving-bed system and were reported in the previous milestone report.

Experimentation

In order to prepare samples for microwave regeneration studies, a large batch of the activated carbon was saturated with NO. The granulated activated carbon (GAC) was saturated to 100% with an inlet NOx concentration of 1000 ppm and a superficial velocity of 25 ft/min. On average, the carbon was capable of adsorbing 27.7 g NO_2/ 100 g of GAC.

Results and Discussion

The ratio of CO and NOx has been determined as a function of input power during regneration. If the NOx in activated carbon is desorbed instead of reacted with carbon, then the ratio of CO and NOx will be much smaller. The following reactions describe the adsorption/ regeneration process:

For adsorption, the entering NO reacts with oxygen to form NO_2 and is adsorbed by the carbon.

1. $\mathbf{NO + \frac{1}{2} O_2 \rightarrow NO_2}$

During regeneration, if the adsorbed NO_2 reacts with the carbon source, some of the carbon will react with the NO_2 to produce carbon monoxide. If this occurs, the carbon is expended each time it is regenerated.

2. $\mathbf{NO_2 + 2C \rightarrow 2\,CO + \frac{1}{2} N_2}$

If during regeneration, the adsorbed NO_2 does not react, but is desorbed, then
the reactor off gas will contain either oxygen, or NO_2. If the gas is desorbed, the carbon is left intact and is not consumed.

3. $\mathbf{NO_2\,(ad) \rightarrow NO(g) + \frac{1}{2} O_2\,(g)}$ **or**
$\mathbf{NO_2\,ad) \rightarrow NO_2\,(g)}$

In preliminary experimentation, 20 SCFH of Nitrogen purge was flowed through a 25-inch quartz tube helix microwave reactor. Approximately, 900 grams of saturated activated carbon were placed in the quartz tube and regenerated with various power levels. The lower the CO/NOx ratio, the more the NO_2 is desorbed, as opposed to reacted. Figure 2 shows that at 1000 Watts, 60% if the NO_2 is desorbed, while 40% is reacted with the saturated GAC. Operating conditions will be altered in subsequent work to try to increase the amount of NO_2 desorbed and decrease the amount of NO_2 that is reacted.

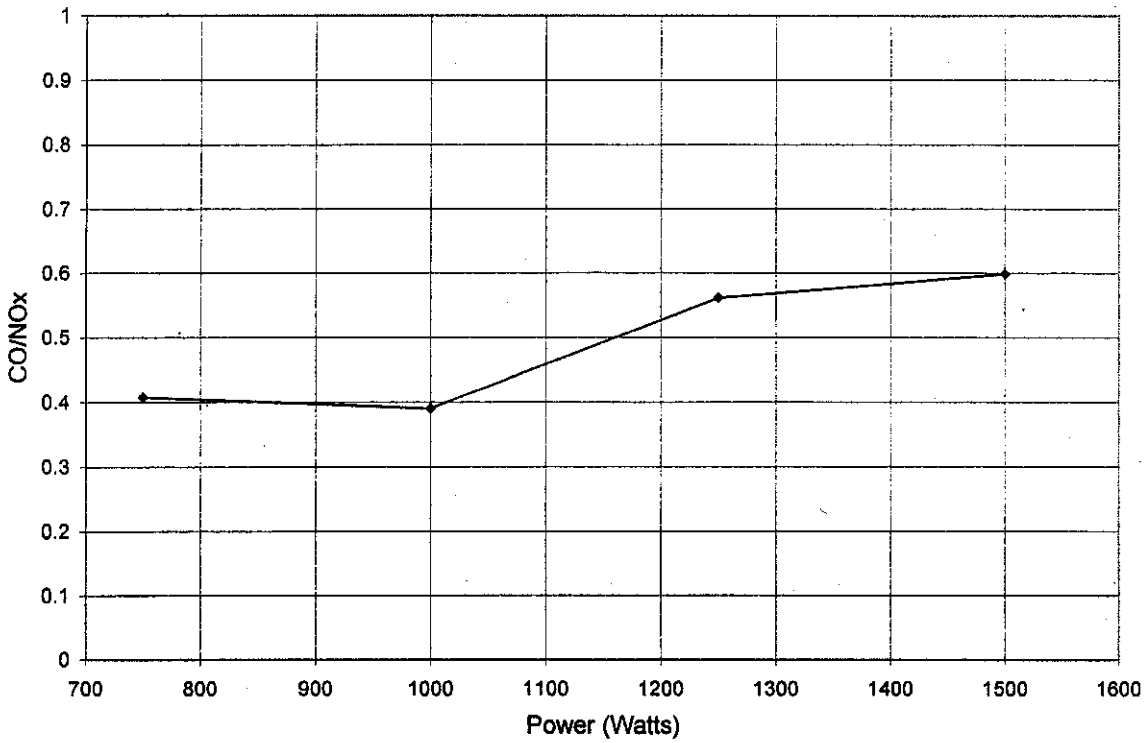


Figure 24: CO/NOx Ratio as a Function of Power

The amount of NO_2 reacted with carbon is dependent on the height of the activated carbon bed. When NO_2 is desorbed, it flows with N_2 through the activated carbon bed. If the flow past the carbon bed height is shortened, the amount of NO_2 reacted with the carbon is reduced. Currently, the purge gas flows from the bottom to the top counter current to the saturated carbon flows. If the purge gas flows from top to bottom, the amount of NO_2 reacted will also be reduced. In the next round of experiments, the purge gas direction will be changed.

These preliminary results are very encouraging. It appears that using the proper reactor arrangement and operating conditions can significantly reduce the amount of NO_2 desorbed from the saturated carbon.

Exhaust Gas Cooler

We worked together with Xchanger Inc. in Hopkins Minnesota to design a heat exchanger that would be appropriate for our needs. A detailed drawing of the heat exchanger is included on page 9. In addition, a detailed specifications sheet is included on page 10. The exhaust gas cooler shown below was designed to cool 120 CFM of gas from 500^{0}F to100 ^{0}F. The heat exchanger is equipped with a variable speed electric motor driven fan to control the cooling capacity of the heat exchanger. The exchanger is outfitted with 2-inch NPT inlet and outlet gas fittings. The unit is made of aluminum and is approximately 2 feet by 2 feet and stands 2 feet tall. The heat exchanger has been installed into the overall process shown in Figure 1.

Experimentation

To find the optimal conditions in which to operate the heat exchanger, preliminary tests were conducted varying the inlet temperature to the NOx adsorber and the speed of the fan. The higher the inlet gas temperature, the lower the activated carbon's capacity to adsorb NOx will be, however, the lower the temperature, the greater the risk of water condensation. It has been determined that 150 ^{0}F is the lowest the inlet gas can be without water condensation. Therefore, inlet temperatures between 150-170 ^{0}F are desirable. Gas was flowed through the heat exchanger at 120 CFM. The results from this testing is presented in the next section.

Results and Discussion

Figure 2 shows that to maintain an adsorber gas temperature of approximately 150 ^{0}F, the fan speed must be about 800 RPM.

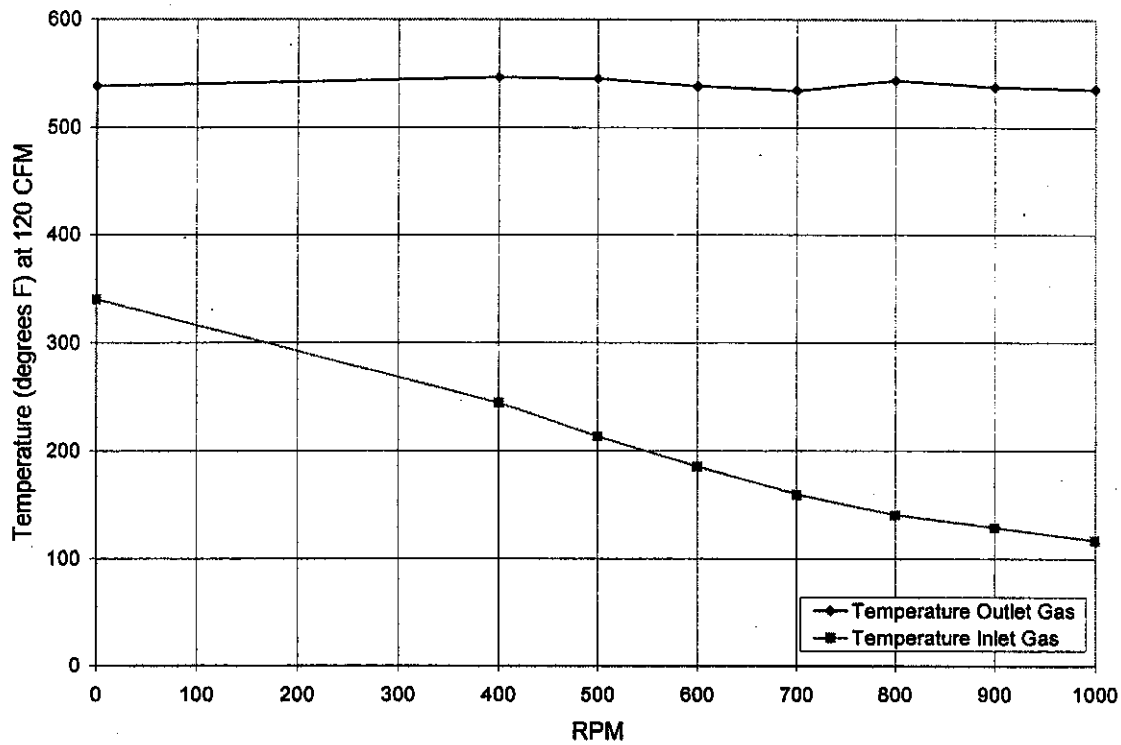


Figure 25: Adsorber Inlet Temperature at 120 CFM as a Function of RPM

Overall System Construction and Shakedown Testing

The construction of the overall system shown in Figure 1 has been completed and shakedown testing has begun. The initial shakedown tests indicated that the gas distributor caused too high of a pressure drop (35- inches of H_2O). As a result, we decided to take the distributor out from the adsorption system and enlarge the orifice sizes in the distributor from 0.20-inches to 0.3125-inches. In addition, we also doubled the number of orifices in the distributor. After this modification, we re-installed the distributor and measured the pressure drop. The new pressure drop was 3–4 inches of H_2O, which is in an acceptable range. It is necessary to maintain the total system pressure drop less than 45-inches of H_2O to protect the diesel engine. If the manifold pressure rises above 45-inches of H_2O, the efficiency of the engine is severely effected.

In addition, we installed two parallel 6-inch monolith filters. These filters offer an initial pressure drop of 12-inches. However, as soot is accumulated on the filter, the pressure drop continuously increases. Due to the limit in the overall process pressure drop, we can allow a maximum pressure drop of 20-inches of H_2O across the filters. Therefore, when the filter pressure drop reaches 20-inches of H_2O, it is necessary to regenerate. The filters that are in place in our prototype system have been regenerated twice and the pressure drop has been decreased from 20-inches H_2O to 12-inches of H_2O. We will continuously investigate better methods of regenerating the filters to reduce regeneration time.

Appendix G: Task 7: Test Heat Exchanger and NOx Adsorption

Regeneration of Saturated Activated Carbon

3930 grams of fresh GAC 8/16 mesh was saturated with 1000 ppm NOx in air in a 4 ¾ inch x 20-inch adsorber. The saturated GAC was then was carefully homogenized and portions of 110 grams were placed in a microwave reactor to study the amount of NOx that was desorbed versus the amount of NOx that is reacted. The amount of NOx that is initially desorbed from the carbon was above the detection limit of the NOx analyzer, 10,000 ppm, and a 450 SCFH of dilution air was added to dilute the desorbed NOx stream. A 10 SCFH purge gas flow of Nitrogen was used to sweep the desorbing NOx from the reactor. Various power levels were tested on a batch basis..

Results and Discussion

The amount of CO and NOx in the reactor outlet stream shows how much NOx is desorbed and how much is reacted with carbon. Figure 1 shows the CO concentration in the outlet stream as a function of time and power level. Higher CO levels correspond to higher microwave power levels. Carbon monoxide is a clear indicator of the NOx reaction with the carbon. We use CO data to determine the extent of NOx reaction with carbon. To conserve the activation carbon, we need to minimize the CO production.

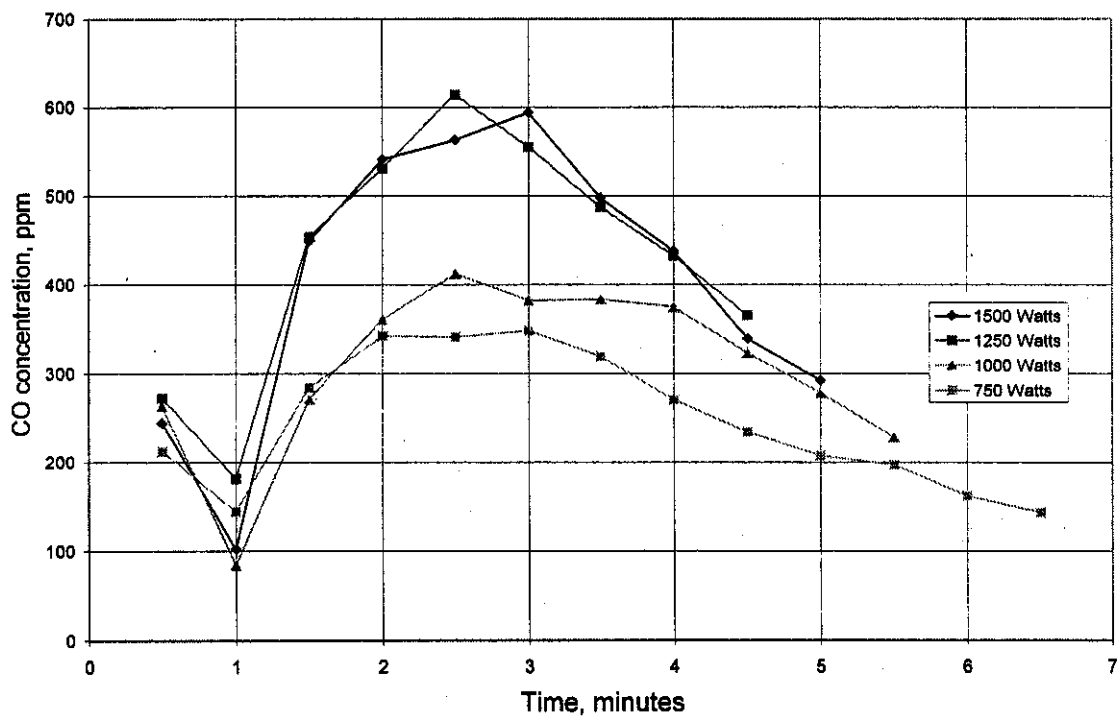


Figure 26: CO Concentration as a Function of Time

Figure 2 shows corresponding NOx concentration as a function of time and microwave power level. NOx responds quickly to microwave introduction and desorbs almost immediately.

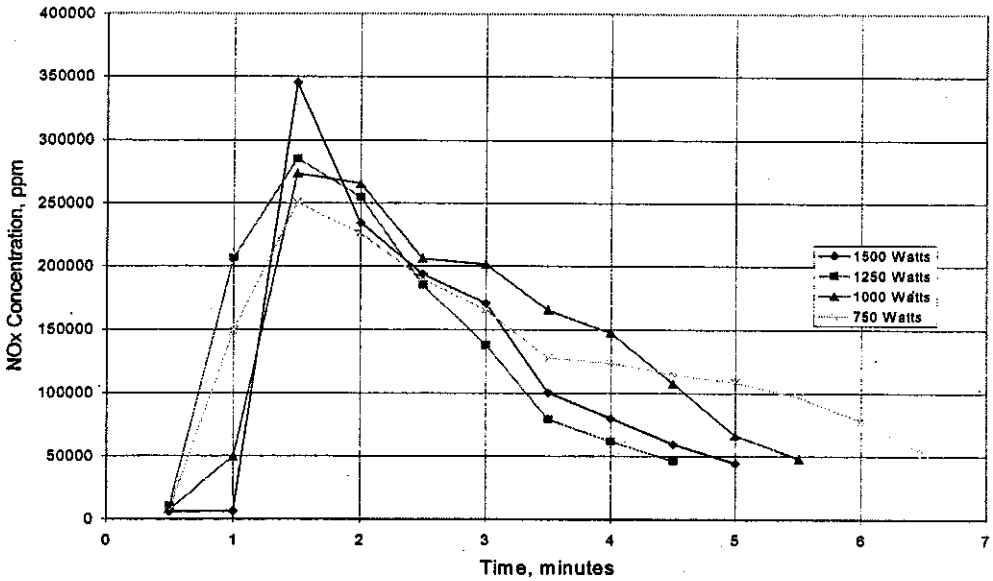


Figure 27: NOx Concentration as a Function of Time and Power

The amount of NOx that is desorbed, as opposed to reacted, can be determined from the CO/ NOx ratio. When NOx reacts with the carbon, the CO is produced. Therefore, the smaller this ratio, the more NOx is desorbed, instead of reacted. Figure 3 shows this CO/ NOx ratio as a function of microwave power. Higher microwave power levels corresponds to larger CO /NOx ratios. This is expected because microwave power drives the NOx reaction with the carbon. Figure 3 clearly shows that less than 1% of the NOx reacts with carbon when the activated carbon is used instead of char as an adsorbent. When the saturated char is regenerated by microwave energy, more than 80% of the NOx reacts with the carbon. Therefore, it was decided to use the activated carbon as indicated in our previous milestone report.

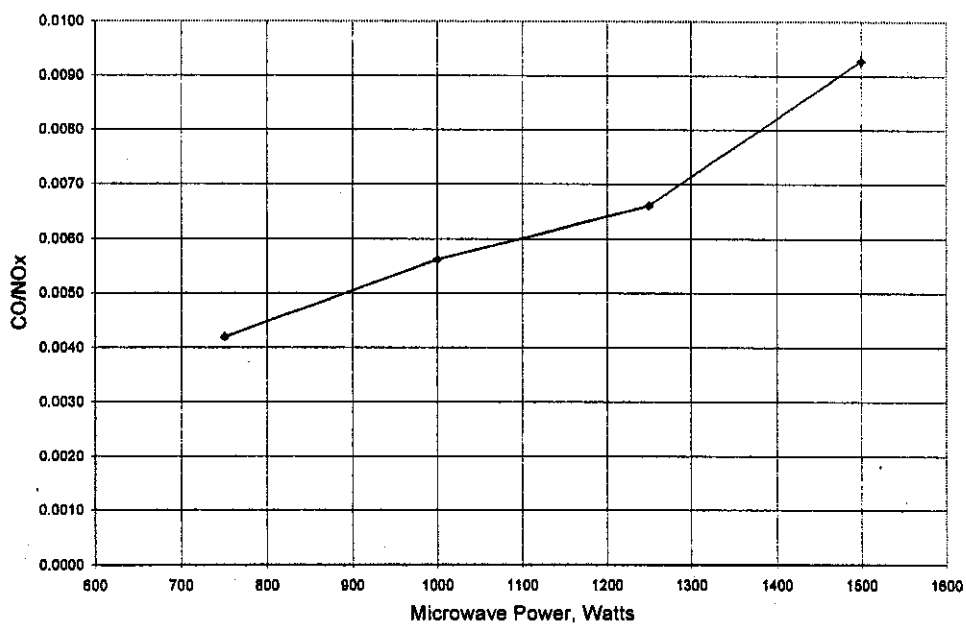


Figure 28: CO/ NOx Ratio as a Function of Microwave Power

Shakedown Testing Activities

The integrated system has been completed and several shakedown and startup cycles have been performed. Long-term duration testing has begun. The following table 1 summarizes the key problems encountered during shakedown and the corrective actions taken. Char has been used as an adsorbent and will be used until the shakedown tests of the integrated system are completed. However, we will replace the char with activated carbon when all shakedown tests are completed.

Problem Area	Description	Solution
Pressure drop across the outlet manifold.	5-inches water pressure in top of adsorber bed which would not allow the carbon to feed from the upper hopper.	Increased the diameter of the piping from one 2-inch outlet to two 4-inch outlet manifolds.
Fines overhead	Significant dust cloud comprised of sub-micron particles during carbon transfer.	A cyclone was constructed and added. After the cyclone, an automotive air cleaner filter with a thick foam rubber pad wet with oil was added. All air-handling joints and connections were sealed with silicon sealant.
Inlet manifold	Too much back pressure on the diesel power source.	Increased openings from 0.2-inches to 0.321-inches. Doubled the number of holes in the distribution manifold.
Electric Valve Actuators	Not functioning properly, sporadic opening and partial closing	After changing the micro switches, making numerous adjustments, it was determined that the actuators were overheating due to excessive duty cycling and resulting heat. Installation cooling fans on actuator housing which sufficiently cooled the actuators. Changed from 250-inch-lbf actuators to 700-inch-lbf actuators.

Table 5: Shakedown Activities

Long-term Durability Testing

Durability testing has started and data representing the first several hours of operating are presented in Figures 4-7. The CO levels remained constant at approximately 200 ppm, while the oxygen remained constant at 14 percent. In addition, the manifold temperature fluctuated between 500-550 degrees Fahrenheit. The temperature of the gas entering the absorber was also constant at 149 degrees Fahrenheit. Figure 4 shows the pressure drop across the monolith soot filters as a function of time. The periodic lowering of this pressure drop represents a regeneration cycle. This regeneration is also reflected in the engine manifold pressure shown in Figure 5. Figure 6 illustrates NOx concentrations entering the bed and corresponding exit gas NOx concentration. The downward trend of the inlet NOx indicates that the bed is deteriorating. Figure 7 shows the NOx removal efficiency. These early data show only 50% NOx removal. We anticipate this improving after bed regeneration. In addition, after approximately 15 hours of operation, the NOx removal efficiency was very low. It was then discovered that the carbon bed was only 12 inches deep. We also discovered the microwave power was not sufficient (<1000 W). The bed was then increased to 22 inches and magnetrons were replaced to produce adequate microwave energy (1500 W). Operation results are forthcoming.

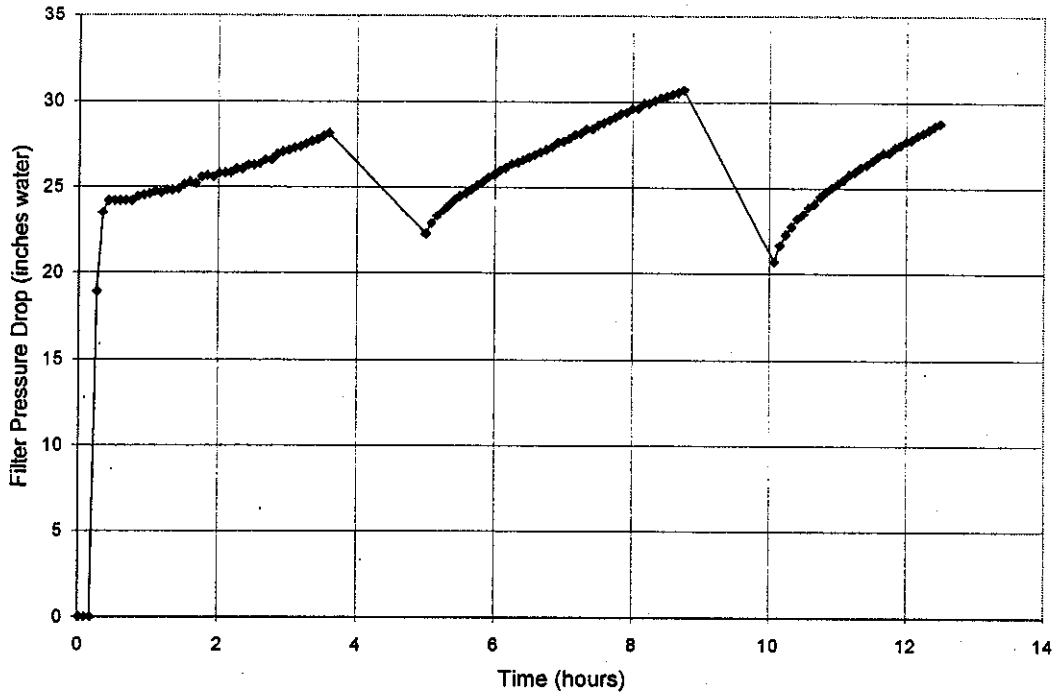


Figure 4: Pressure Drop Across the Soot Filter as a Function of Time

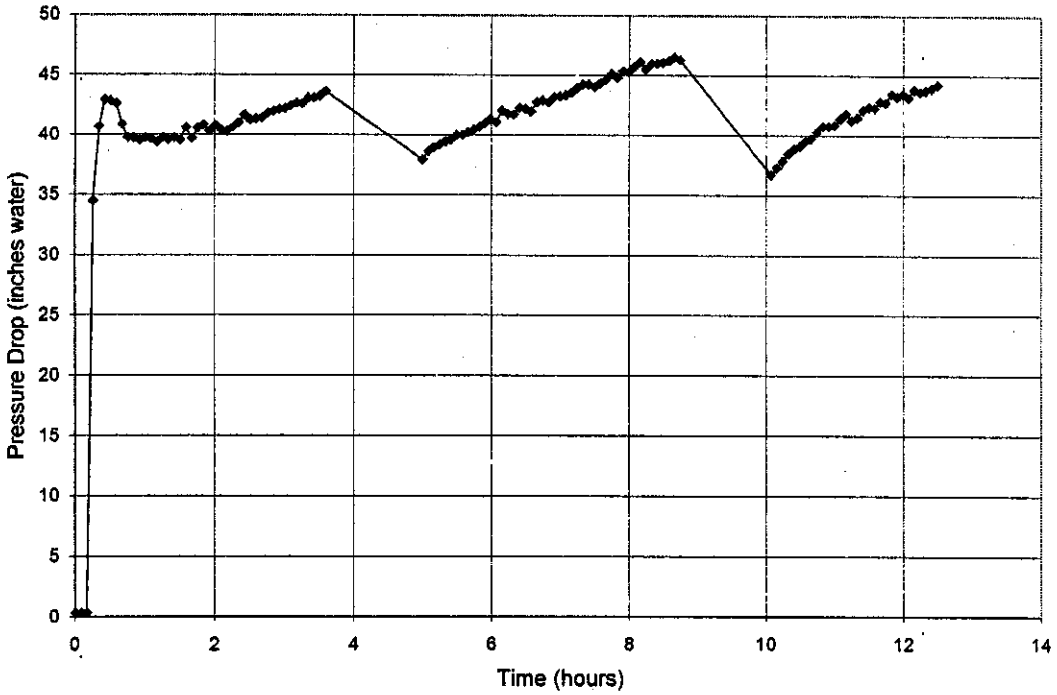


Figure 5: Pressure Drop Across the Engine as a Function of Time

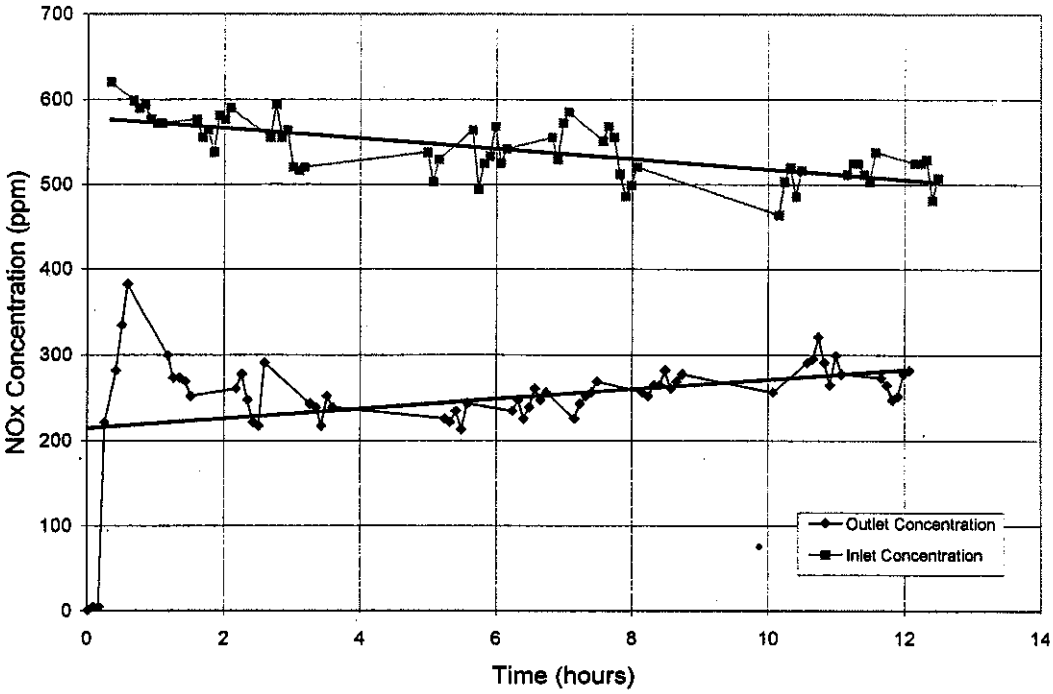


Figure 6: NOx Concentration as a Function of Time

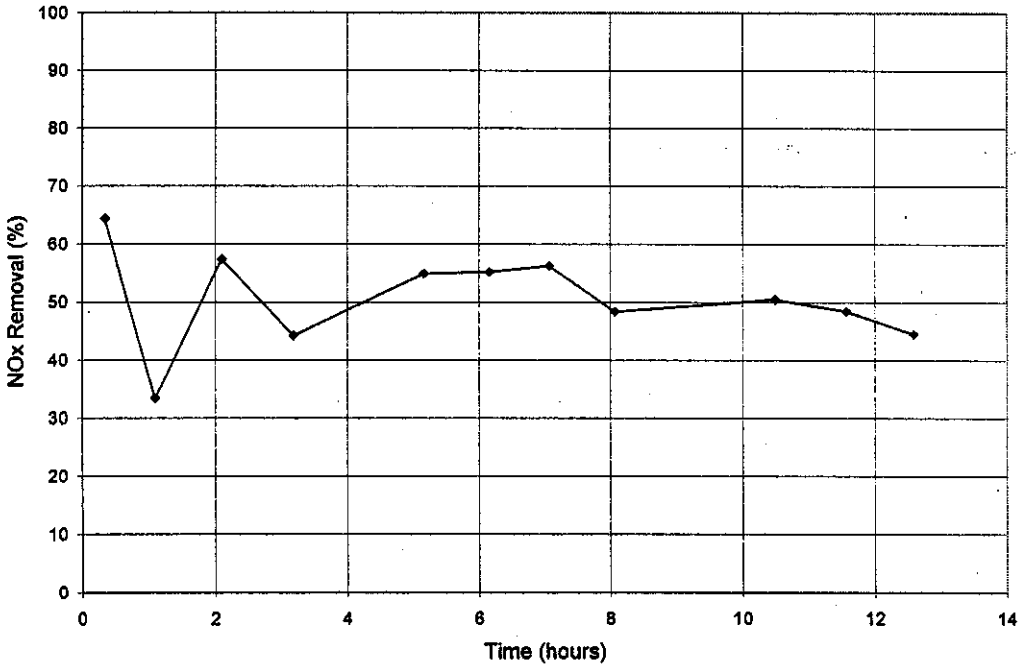


Figure 7: NOx Removal Efficiency as a Function of Time

Appendix H: Task 8: Study Gas Residence Time and Particle Size

Adsorbent Particle Size Testing

Three different carbon products have been tested so far. A fourth product is currently being tested. Each of these adsorbent materials has a different grain size. The three adsorbents tested so far are: crushed ¼-inch briquettes, 10 mesh calcined char, and 6 to 10 mesh granular activated carbon (GAC 6-10). We are currently testing a four-millimeter diameter activated carbon pellet material. With each of the carbon products, the minimum residence time is in the range of 2 seconds with little variation between products.

Crushed Calcined Char Briquettes

The FMC Corporation in Kemmerer, WY produces 1-1/2 inch briquettes of calcined char bonded with polymerized coal bitumen. We received about 1000 pounds of this material crushed to about ¾-inch to 1/8-inch in size. We separated out enough ½-inch material to fill the contactor to 38-inches and made a series of NOx adsorption runs using raw diesel exhaust gas carrying 1000 ppm of NOx. NOx breakthrough was almost immediate and total NOx capacity was poor at less than one percent by weight. Significant by-passing and the immediate breakthrough at 200 ppm NOx was also noted with a residence time of 1.9 seconds. Pressure drop was very good at 3 inches water for the 38-inch bed used. Near complete saturation of the char bed occurred within 4 hours where the outlet NOx concentration exceeded 700 ppm. It was determined that residence times as high as 3 seconds did not improve the char's NOx adsorbing performance. We also found that the 1/2-inch material was difficult to regenerate. Microwave power, 1500 Watts, applied to the crushed briquettes passing through the regeneration reactor at 30-35 lbs/hr required 4 passes to regain approximately half of the NOx capacity measured in the original material. Attrition percentage was very low at 0.5% per pneumatic cycle.

10-mesh Calcined Char

We also experimented with 10-mesh calcined char, also an FMC product. The calcined char is the carbonaceous feed stock taken before briquetting. Therefore, it is free of the bituminous binder. The surface area of the calcined char is between 100-150 m^2/g, as opposed to the briquettes that have a surface area of approximately 50 m^2/g. The 10-mesh char was placed in the same absorber and the bed height was 36 inches. Raw diesel exhaust was passed through the bed carrying 1000 ppm of NOx. We did not observe any by-pass in the bed and a bed pressure differential of 8-10-inches of H_2O was measured with a gas residence time of 2-seconds. Initial NOx breakthrough was observed within one hour and the bed was completely saturated within 6 hours. Microwave regeneration was achieved at 1500 lbs/hr and 1200 Watts of applied power. However, the capacity of the char to absorb NOx is low at less than 2% by-weight absorption capacity. A residence time of up to 3 seconds did not significantly improve the char performance. The attrition rate of the calcined char was approximately 1%.

Granular Activated Carbon

The final material tested was Calgon F 6-10 granulated activated carbon (GAC). This is a commercially available activated carbon made from sub-bituminous coal specifically for automotive applications. The absorber bed height was 32 inches. Breakthrough was observed in eight hours and good NOx capacity was measured at 6% by weight. In addition, the GAC regenerated easily with 600 Watts applied microwave power at a feed rate of 15 lbs/ hr. through the reactor. Complete NOx saturation was observed after 36 hours of run time. This material was the most favorable for NOx removal of any material used. However, the mechanical strength of the GAC is significantly less than the char products. A large amount of fines was produced during these runs. The attrition rate was 5% per transfer and the fines were very difficult to remove from the pneumatic vents because of the fine powder consistency of the fines.

Currently, we are scheduled to test one additional carbon material from Filtration Media Group in Union Gap, Washington. We have received 750 lbs of activated carbon pellets numbered 604, that are 4-mm in diameter and 8 to 10 mm in length. These pellets have a surface area of 1000 m^2/g. We have started testing on this material and attrition is less than 0.1%. After one hour of NOx absorption testing, there is no measurable NOx leaving the 38-inch bed. Additional test results will be included in our next report.

Durability Testing

During this testing period, the prototype system ran for periods of 24 to 48 hours to determine if there would be any operational problems. A number of changes were make in the integrated system. The changes resulting from these runs are shown in Figure 1.

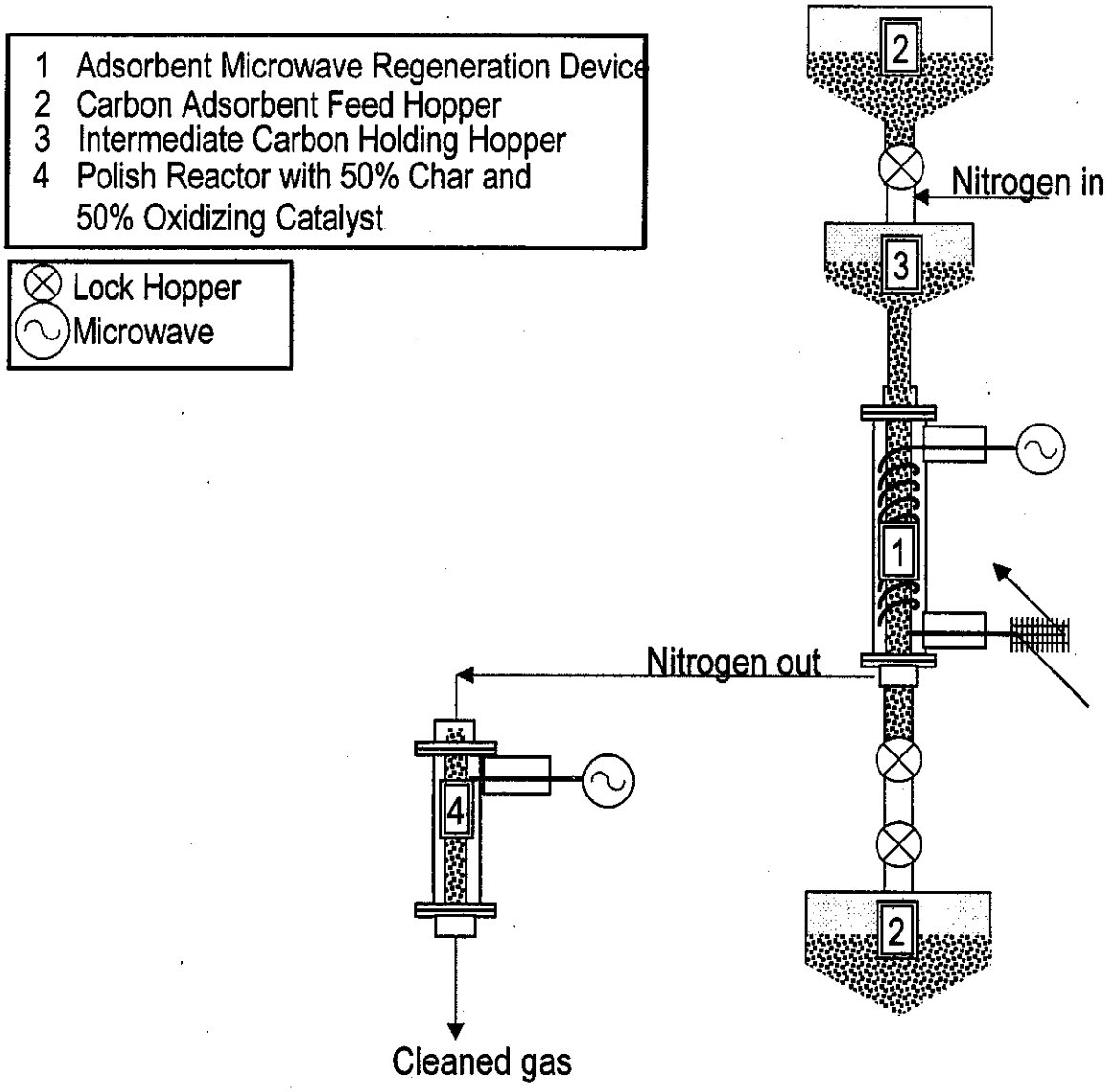


Figure 29: Newly Re-configured Regeneration Prototype

Figure 1 shows the changes made to the regeneration unit. Previously, the nitrogen purge gas was introduced at the top of the microwave regeneration device (1). The nitrogen gas, along with water vapor from the reactor, would escape into the carbon feed hopper (2) when the feed valve was opened causing both the loss of the nitrogen and the contamination of the feed carbon with condensing water vapor. During long term run testing we discovered that the feed carbon would bridge occasionally interrupting the flow of carbon into the reactor. To reduce the frequency of the feed valve operation, we added an intermediate feed hopper to store the feed carbon for a longer period of time between feed valve operations and moved the nitrogen addition point to the top of this new hopper. This change eliminated the feed carbon bridging problems. The nitrogen now continuously exits through the bottom of the

microwave reactor and sweeps gas through the polish reactor. The polish reactor is packed with eight inches of calcined char and eight inches of reducing catalyst coated onto 1/8-inch alumina beads impregnated with 25% 800 mesh SiC. The char and catalyst work together to decompose any NOx leaving the main reactor

The second major problem encountered was the clogging of the reactor outlet nitrogen fitting with carbon fines. A 1.5 inch tee was added that equipped with both a screen and a short section of glass fiber. The glass fiber and screen prevent carbon fines from entering the nitrogen purge line.

One minor adjustment was made to the vacuum head. In the vacuum filter is off-center and the carbon is introduced tangentially. Cloth filters were too easily clogged with fines and were replaced with a foam rubber filter. When the carbon particles entered the hopper, they would strike the filter body and wear a hole in the filter material. A small piece of polypropylene baffle was added to the carbon entry port to protect the filter from wear.

The activated carbon runs gave problems with very fine particles generated during pneumatic transfer of the adsorbent. The fines quickly plugged the final filter and some fines would leak into the room during transfer. To address this problem, we changed the vacuum head run times from simultaneous to staggered operation. Since there is only one cyclone, we added two check valves to the vacuum lines to prevent leakage of fines from one vacuum head to the other during staggered running. This change almost eliminated the fines from the activated carbon.

2.0 Future Work

In the final reporting period, we will finish our shakedown testing and transport the prototype for an on-site demonstration at McClellen Air Force Base in Sacramento, CA.

Appendix I Task 9: Demonstration, Technical and Economic Evaluation and Final Report

On-Site Demonstration

Figure 1 presents a process flow diagram of the entire demonstration prototype. The diesel exhaust flows from the engine into a microwave-based soot filter where approximately 90% of the soot is removed. The gas then flows through a bed of oxidation catalyst (catalyst converter) where VOCs and CO are converted to CO_2 and H_2O. The gas is then passed through a heat exchanger where the gas is cooled to better facilitate the adsorption of NOx by the carbon adsorbent. The cooled gas enters the carbon adsorbent bed where the NOx is removed. Clean gas then exits through the top of the adsorbent bed. Spent adsorbent continuously passes into a storage hopper located at the base of the adsorber and is periodically transferred to the regeneration feed hopper. The saturated activated carbon is regenerated with 2450 MHz microwave energy while passing through the microwave reactor. The second microwave reactor is purged with 5 SCFH nitrogen to prevent the air leakage into the reactor. The regenerated carbon is stored in a hopper located below the regeneration reactor. The regenerated carbon cycles back into the adsorber feed hopper where it awaits the next round of NOx adsorption. The NOx desorbed from the carbon flows into the second microwave reactor and is decomposed to N_2 and O_2 by the reducing catalyst. The gas from the second microwave reactor joins with the inlet gas.

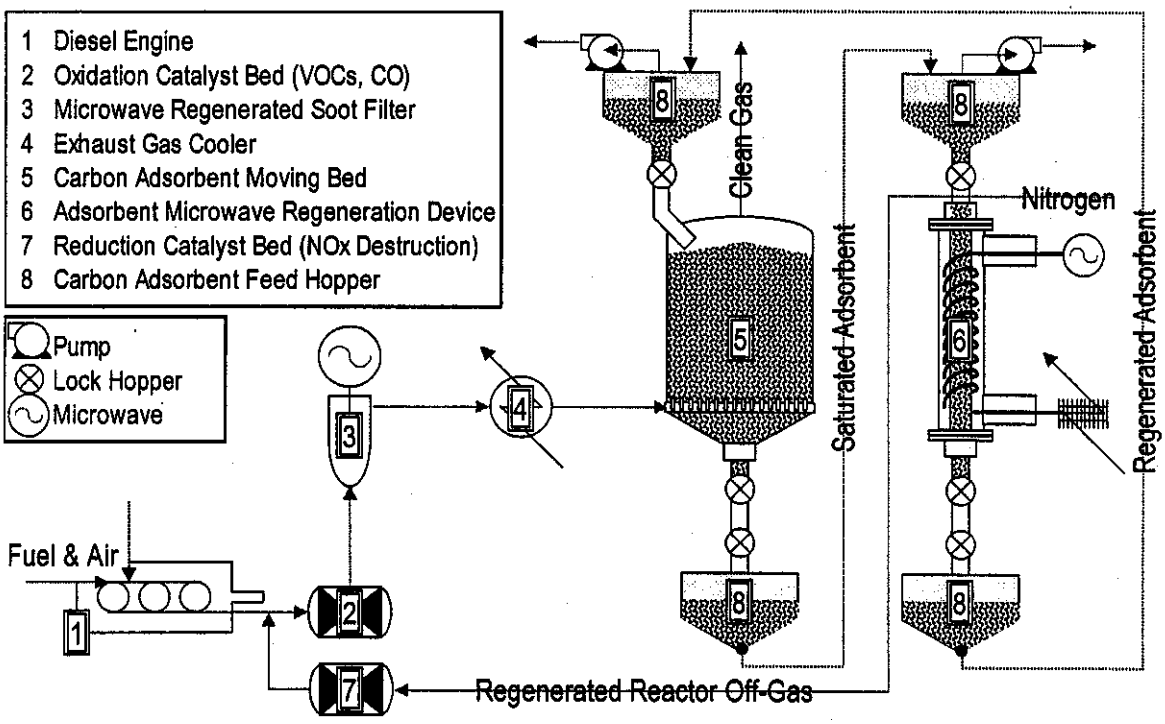


Figure 30: Process Flow Diagram for CHA NOx Process

Figure 2 shows the overall picture of the entire demonstration prototype that was set-up at the McClellan Air Force Base.

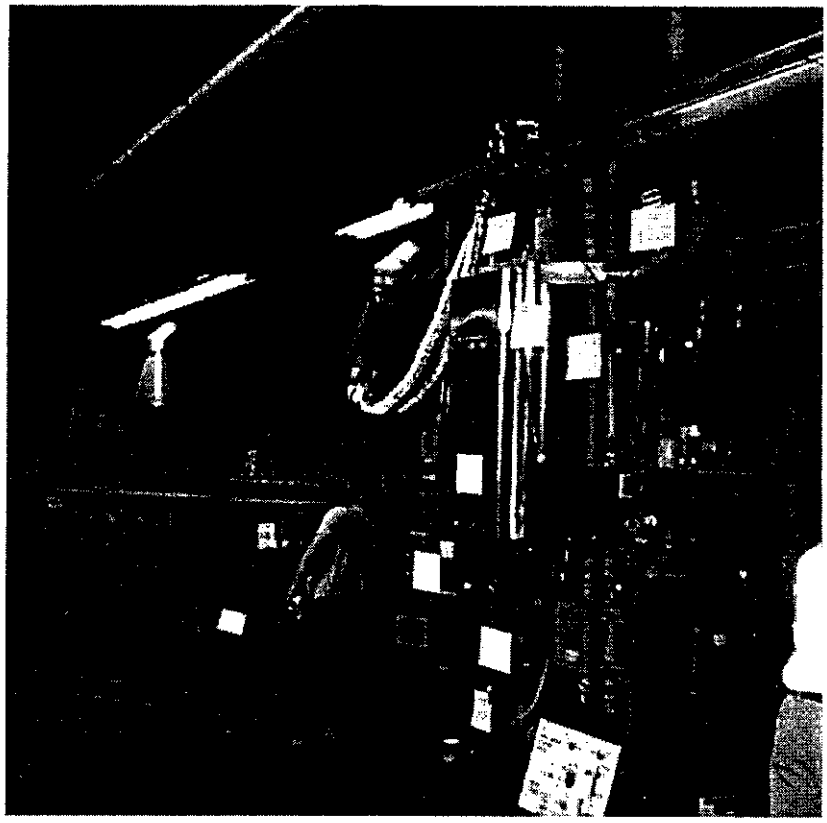


Figure 31: CHA Diesel Exhaust Treatment Prototype

Figure 3 shows the stationary diesel engine used for the prototype on-site demonstration. The engine produced 147 CFM of diesel exhaust. The average NOx production from the stationary engine with a 100% load was 1040 ppm. The engine manifold back pressure was an average of 40 inches H_2O.

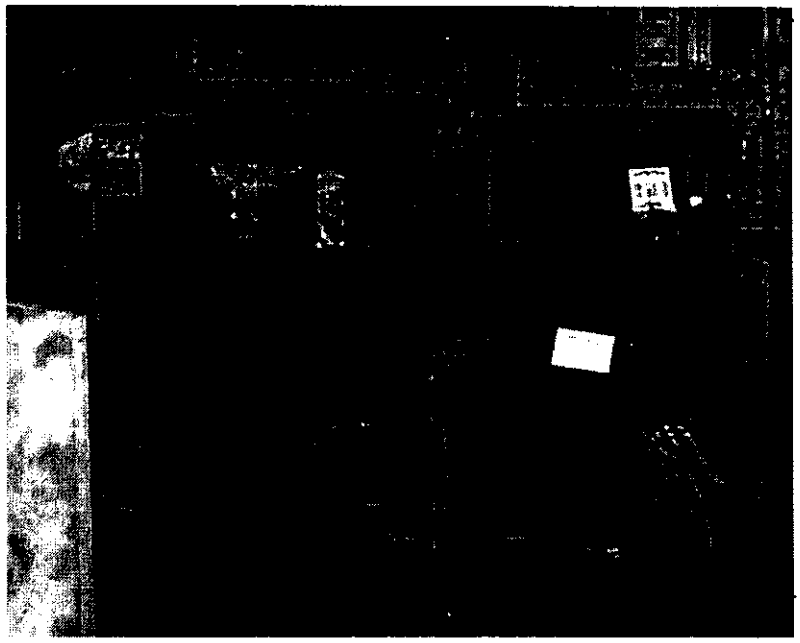


Figure 32: Diesel Engine Used in On-Site Demonstration

Figure 4 shows the piping from the diesel engine to the inlet of the soot filters. The piping is wrapped in fiberglass insulation to minimize heat loss. The incoming diesel exhaust gas temperatures are typically between 600-700 °F immediately after leaving the engine. The gas then flows through the catalyst converter that is located between the soot filter and heat exchanger (not clearly shown in Figure 4). Before the gas enters into the carbon adsorber, it encounters a cooler and is cooled to a temperature between 130-140 °F. Without cooling the gas, the adsorption capacity of the granulated activated carbon is severely reduced.

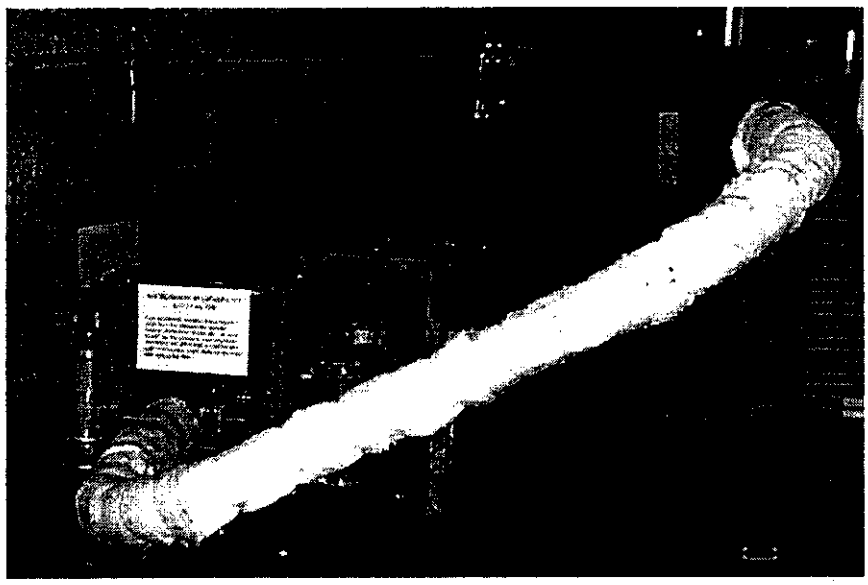


Figure 33: Exhaust Pipe from the Diesel Engine to the Inlet of the Soot Filter

Figure 5 shows the heat exchanger and soot filter used during the demonstration. The heat exchanger was custom designed by Xchanger, Inc. in Hopkins, Minnesota. The heat exchanger is designed to cool 120 CFM exhaust gas from 500 °F to100 °F. The heat exchanger is equipped with a variable speed electric motor driven fan to control the cooling capacity of the heat exchanger. The unit is made of aluminum and is 2 feet by 2 feet wide and stands approximately 2 feet high.

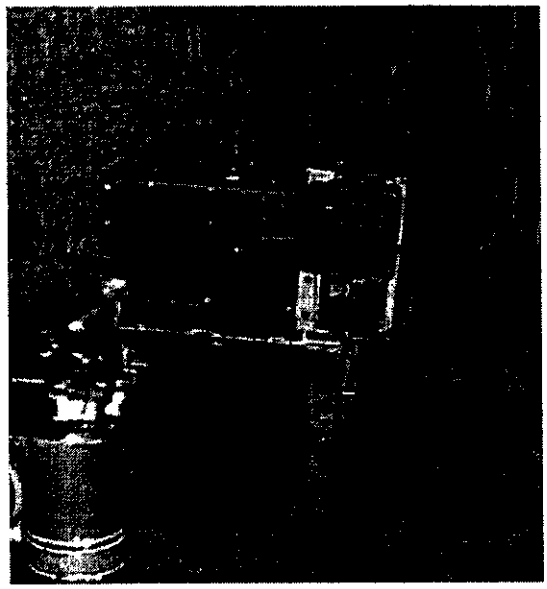


Figure 34: Heat Exchanger used During Demonstration

All other components are shown in Figure 2. Unfortunately, due to poor lighting, pictures of other components are not available.

On-Site Demonstration Tests of Prototype Unit

On 27 February 1998, two representatives from CHA Corporation, Charlie Carlisle and Bob Guffey, traveled to Sacramento with the CHA diesel exhaust treatment prototype in order to carry out an on-site demonstration at McClellan Air Force Base. By the end of Monday, 2 March, the demo unit was completely assembled and ready for testing. A 30 kW diesel generator was positioned near the prototype unit and piping connections were made. On Tuesday, 3 March, a load bank was connected to the generator set and a series of shakedown runs were completed. During the initial shakedown period, some concern arose because the pressure drop across the soot filter unit elevated from 14 inches H_2O to 45 inches H_2O in 20 minutes of loaded run time. This was unusual because typically, the engine start pressure was 10 inches H_2O with an elevation to 24 inches H_2O after eight hours of loaded run time. The McClellan 30 kW unit was powered by a six cylinder White engine that produced 73 hp and 148 CFM of exhaust gas. The McClellan engine produced about 40 CFM more gas than the 58 hp, 35 kW test engine used at CHA Corporation. In order to proceed with demonstration runs, one of the filter elements was removed. Wednesday, 4 March was the first day that the prototype was demonstrated for various visitors.

Also on Wednesday, a newer generator set was moved in and attached to the demo unit. The first engine used was worn out significantly and the soot volume was unusually high. The maximum NOx concentration in the exhaust gas of the first engine was only 375 ppm. The newer engine had fewer hours of run time and generated considerably less soot. The NOx concentration in the exhaust gas of the newer engine was between 1000 and 1100 ppm when loaded. The balance of runs was carried out using the newer generator set. Runs were continued throughout Wednesday, Thursday and Friday. The total run time on the unit was 9.2 hours. The outlet gas did not contain any measurable NOx indicating that 100% of the NOx was removed in the adsorber. The following is typical test conditions for the demonstration runs:

Typical Conditions for Test Runs

Exhaust gas temperature at filters	650 °F.
Exhaust gas temperature after cooler	137 °F.
Gas flow rate from engine	147 CFM
Engine Load	100%
Exhaust gas NOx concentration	1040 ppm
NOx concentration leaving unit	zero ppm
Engine manifold pressure	40 inches H_2O
Test room temperature	65 °F.
Carbon feed rate	15 lbs/ hr
Regeneration microwave power	1600 Watts

The following is a list of visitors who came to see the prototype demonstration:

Visitor	Representing
Tim Chapman	BDM Federal
Craig Burnett	McClellan AFB
Bert Gross	McClellan AFB
John Carroz	McClellan AFM
Capt. Paul Simonich	McClellan AFB
Col. Adair	McClellan AFB
Kelly Hughes	Stationary Source Division, CARB
David Mallory	Stationary Source Division CARB
Laura Zaremba	Stationary Source Division, CARB
Don Koberlein	Stationary Source Division, CARB
Ralph Propper	Research Division, CARB
Hector Maldonado	Research Division, CARB
Robert Grant	Research Division, CARB
Manjit Ahuja	Research Division, CARB

Bob Barham	Research Division, CARB
Randy Pasek	Research Division, CARB
Marla Mueller	CARB—Project Manager
Paul Tower	Filter Media Group
Dave Boyd	West General Associates
Max Jones	TENCO Catapillar
Harry Bristow	TENCO Catapillar
Kent Lindberg	TENCO Catapillar
Alan Suleiman	SMUD
Lamar Mitchell	MAQMD
Rick Wiesner	SMUD
Phil Mook	McClellan AFB
Mark Hall	McClellan AFB
John David	McClellan AFB
Grant Chin	CARB
Mike Tollstrup	CARB

Technical and Economic Evaluation

A prototype unit for treating 100 SCFM of diesel exhaust gas produced from a 35 kW (58 hp) diesel engine was successfully demonstrated. However, because of the size of the unit, it would be more economical to combine the exhaust gases produced by ten 35 kW diesel engines. Therefore, we estimated the capital and operating costs for a 1,000 SCFM, or 250 kW (314 hp), diesel engine using the test data obtained from the prototype unit. The 250 kW diesel engine produces 617 SCFM of exhaust gas.

A typical exhaust gas produced from a 250 kW diesel engine with a 100% load has the following composition (EPA Report # EPA-453/R-93-032, July 1998):

	kg/hr	Volume %
Carbon Dioxide	158.57	8.14
Nitrogen	938.28	75.69
Oxygen	112.43	7.94
Water	62.74	7.87
Carbon Monoxide	0.91	0.08
NO	1.70	0.13
NOx	2.60	0.12
Sulfur Dioxide	0.20	0.20
Hydrocarbons	0.30	
Diesel Particulate Matter	0.012	

The data is based upon steady state operating conditions consisting of a temperature of 85 °F, a pressure of 28.38 inches Hg, and No. 2 diesel fuel. In addition, the NOx shown is not present in the exhaust. It is based on the assumption that the NO is converted into NOx in the atmosphere.

Technical Evaluation

The CHA Diesel Exhaust Treatment System was based on the use of activated carbon and microwave energy. The prototype treatment system was designed, fabricated, and operated for the cleanup of diesel exhaust gas generated from a 58 hp diesel powered electricity generator. The prototype unit was successfully demonstrated at the McClellan Air Force Base in Sacramento, CA. The prototype is a stand alone unit but is not on wheels and is larger than the existing power carts. Although more than 90% of NOx and VOCs are removed, the unit is not portable and is therefore not applicable for use with power carts. The unit is more applicable for treating the diesel exhaust gases produced by standby generators. The prototype can easily be scaled up for use with diesel engines greater than 58 hp.

Soot Removal

The ceramic monolith filter has a collection efficiency of approximately 90%. Two filters (5 5/8 inches in diameter and 6 inches long) were installed to the prototype unit. The pressure drop was acceptable for approximately 8 hours

operation. Every 8-hour operation, the filters were successfully regenerated with microwave energy. For larger units, a 12-inch diameter monolith filter can be used. A magnetron, conceptually similar to those used in home microwave ovens, is installed in the monolith filter. The microwave-based ceramic monolith filters were regenerated more than 50 times. After each regeneration with microwave energy, the pressure drop across the filter returned to approximately 10 inches of water with each filter flow rate of 60 CFM.

The microwave regeneration of ceramic monolith filters can be completed within one hour and has been repeated more than 50 times. Therefore, a combination of a bigger sized (a 12-inch diameter monolith is currently available) monolith and a larger number of filters can be easily adopted to treat a larger exhaust gas flow rate. The microwave-based filter has a strong possibility to be used for the underground mine diesel engines.

NOx Removal

Both char and activated carbon were tested to remove NOx from diesel exhaust gas. During microwave regeneration, NOx reacts with char to produce CO and N_2, but easily desorbs from the activated carbon. As a result, char is consumed but the activated carbon is not consumed. Furthermore, the NOx removal efficiency is greater with the activated carbon than with char. Recently, we found activated carbon pellets manufactured by the Filtration and Media Group, Inc of Union Gap, Washington. These carbon pellets produce a negligible amount of dust during transportation. This carbon pellet was used for demonstration at the McClellan Air Force Base. Therefore, it was decided that the activated carbon pellet would be used in commercial operation.

The vertical adsorber is used for the prototype unit. This type of adsorber is applicable for gas flow rates up to 1000 CFM. For gas flow rates greater than 1000 CFM, a radial type adsorber should be used to reduce the size of the adsorber. Sorbent Control Technologies, Inc in Elgin, Illinois, designed and manufactures a radial adsorber that can be easily adapted for the NOx removal system.

The activated carbon adsorber removed more than 90% of the NOx in the diesel exhaust gas and did not have any problems with frequent shutdown and start up operations.

Microwave Regeneration of Saturation Carbon

The saturated activated carbon was continuously regenerated in the microwave reactor at a rate of 17-20 lbs/hr (8-9 kg/hr). The NOx desorbed from the activated carbon pellets was decomposed to N_2 and O_2 by an oxidation/ reduction three way catalyst in the second microwave reactor. The catalyst contains silicon carbide that absorbs microwave energy. A 2.31-inch diameter quartz tube was utilized in the microwave reactor for the 100 CFM prototype unit. For a larger flow rate, a belt conveyor microwave reactor has been built and tested for the microwave regeneration of saturated carbon. A vertical moving bed reactor can be used for larger units if a 915 MHz microwave generator is used in place of 2450 MHz magnetrons. The use of magnetrons makes the microwave reactor much cheaper than using the microwave generators. Because of the cost and flexibility of using 2450 MHz magnetrons, a belt conveyor microwave reactor is recommended for larger units.

The microwave regeneration of saturated carbon was successfully repeated during the operation of the prototype unit. Therefore, it will not be difficult to scale up the microwave reactors for regeneration of saturated carbon and destruction of NOx desorbed during regeneration.

Pressure Drop and Exhaust Gas Cooler

The maximum system pressure drop did not exceed 45-inches H_2O. The major pressure drop occurred at the soot filters (20-30 inches H_2O). The exhaust gas cooler manufactured by Xchanger, Inc. in Hopkins, Minnesota worked well to cool 120 CFM of exhaust gas from 500 °F to 100 °F. The heat exchanger is equipped with a variable speed electric motor driven fan to control the cooling capacity of the heat exchanger. The scale-up of the heat exchanger will not be difficult.

In general, the prototype unit was successfully used to demonstrate the various steps of the CHA Diesel Exhaust Treatment System. The system can easily be scaled up to remove NOx and VOCs from the various gaseous effluents.

Economic Evaluation

An economic evaluation was prepared based upon components used in the prototype unit. In addition, operating costs were estimated for treating 1000 SCFM of diesel exhaust gas containing NOx, CO, VOCs, Hydrocarbons, and diesel particulate matter (DPM).

Process Description

Figure 1, previously presented, shows an overall process diagram for the treatment of stationary diesel exhaust gas. The system represents a continuous process of adsorption and regeneration through which diesel exhaust gas can be continuously cleaned. The saturated carbon adsorbent is passed through a regeneration column where microwaves are used to regenerate the carbon. Diesel exhaust flows into the carbon adsorber at 150 °F and 1,000 SCFM. As is apparent in the schematic, diesel exhaust gas first encounters an oxidation catalyst bed that oxidizes and destroys almost 100 percent of the VOCs and CO in the exhaust gas. The gas then flows into a microwave regenerated soot filter where more than 90 percent of the soot is removed. The filter is periodically regenerated. The gas is then routed through a heat exchanger for cooling. The gas is cooled from a temperature of 500 °F to a temperature between 150-200 °F to better facilitate the adsorption of NOx onto the carbon. The gas enters into the activated carbon adsorber where the NOx is adsorbed onto the carbon. The clean gas then exits through the top of the adsorber. The saturated adsorbent passes into a hopper and is then introduced into the 10-inch ID regeneration column or a conveyor belt regeneration reactor. In the regeneration reactor, it is purged with a nitrogen stream to prevent adsorbent oxidation. Once in the regeneration reactor, the NOx laden carbon is exposed to a 915 or 2450 MHz microwave under a nitrogen purge to desorb the NOx and regenerate the carbon adsorbent. The concentrated NOx stream flows into a reduction catalyst bed where the NOx is destroyed. The regenerated adsorbent is then passed into a hopper and cycles back into the adsorbent bed.

Capital Cost Estimate

The purchase cost for the adsorber was estimated by the method described in the textbook Air Pollution Control by David Cooper and F.C. Alley, pg. 441. The other equipment purchase costs were obtained from equipment benders and in-house cost data. The purchase cost for each equipment items is presented in the following table.

Equipment Name	Cost	Description
Adsorber	$13,300	30.5"x30.5"x40"
4-inch Lock Hopper Valves	3 x $1,000 = $3,000	Actuating valves
Air cooler	$5,000	Heat exchanger used to cool the inlet diesel exhaust gas
4 hoppers	4 x $1000 = $4,000	Carbon holding device
Catalytic converter	$300	
Soot filter	$7,500	Monolith Soot Filter
Vacuum pump	$2,000	Used to transport carbon
Controller	$5,000	
Microwave generator and reactor system	$50,000 for 915 MHz or $24,000 for 2450 MHz microwave reactor system	Used for regeneration

Table 6: Estimated Equipment Costs

The total estimated purchased equipment cost is $95,100 when a 915 MHz microwave generator is used for regeneration of saturated carbon. On the other hand, the total purchased equipment cost is reduced to $69,100 when 2450 MHz magnetrons and a conveyor belt microwave reactor for the regeneration are used. The vertical quartz reactor has been operated with 2450 MHz microwaves for a long time without any major problems. However, the penetration depth is only 2-inches, which severely limits the used of a vertical quartz reactor. A 915 MHz microwave reactor will have a penetration depth almost three times greater. Consequently, a 10-inch quartz column can be used as a regeneration reactor if a 915 MHz microwave generator is used. This is the main reasonfor considering a 915 MHz microwave generator for the cost estimate. However, the capital cost is reduced by 27% if 2450 MHz magnetrons are used. A belt conveyor reactor has been constructed and tested using 2450 MHz microwave generator. Therefore, we have chosen a belt conveyor microwave reactor equipped with 2450 MHz magnetrons for regeneration of saturated carbon for capital cost estimate.

Using 5% freight and a 65% installation cost factor, the total installed equipment cost is $117,470. The purchased activated carbon cost was estimated at $4,310. Assuming 5% freight cost, the activated carbon adsorbent cost will be $5,000. The total capital investment was estimated at $122,470. This is equivalent to $490/ kW.

Operating Cost Estimate

The plant is assumed to operate at the treatment rate of 1000 SCFM of diesel exhaust gas containing NOx, CO, VOCs, hydrocarbons, and dry particulate matter (DPM) for 365 days per year. It is also assumed that the plant is operated automatically and controlled by a computer. Hence, no personnel attendance will be required. Assuming that the labor equivalent of 1/4 person will be required to operate the plant, the loaded direct labor cost is $13,000 per year.

Supplies and material costs, including the price of the activated carbon are estimated at $20,000/year. The maintenance cost is assumed to be 3% of the capital investment, or $3,675/year. The annual cost for taxes and insurance are taken as 3% of the capital investment, or $3,675/year. The annual plant power demand is estimated at 65,700 kW*hr and the cost of power is estimated to be $0.045 per kW*hr resulting in an annual cost of $3,000. The total annual operating cost is $43,350. This is equivalent to $1,734/ ton of NOx or $1,117/ ton of pollutants removed.

However, the diesel engine is not used 24 hours a day. The majority of diesel engines are used only approximately 175-2080 hours per year. Therefore, the annual operating cost will be between $866 and $10,293/ year.