

FINAL REPORT

PROJECT NO. A-1073

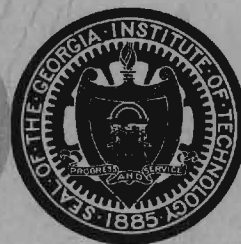
AUTOMOBILE MUFFLER RESEARCH PROGRAM

D. R. HURST

January 1 to October 31, 1968

Performed for
Kleen-Air Corporation
1575 Powder Springs Road
Marietta, Georgia 30060

1968



Engineering Experiment Station
GEORGIA INSTITUTE OF TECHNOLOGY
Atlanta, Georgia

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I. INTRODUCTION

Project work on a catalytic exhaust gas converter was started in January of 1968 when Mr. L. R. Daugherty and Dr. J. H. Henderson of Kleen-Air Corporation brought a prototype exhaust gas converter to the Georgia Tech Engineering Experiment Station to be evaluated for automotive exhausts. The catalyst system was a previously patented (U.S. 1,867,325, Nov. 18, 1929) mixture of iron, chromium, and cerium oxides supported on stainless steel mesh in a muffler housing. Subsequent tests with leaded gasoline showed the oxidation capability of the device to be almost totally inactivated after only a few hours of running on an automobile in normal service. Therefore, a program was initiated to find a suitable catalytic system for both automotive and diesel exhaust systems.

II. REVIEW OF THE LITERATURE

A review of the literature began with an investigation of presently marketed catalytic converter systems. Only one manufacturer, Oxy-Catalyst Company, is marketing with commercial acceptance. Their advertising literature 1/ states that their systems are finely dispersed platinum metal catalyst supported on 1/4" spheres of α -alumina. These systems are designed only for applications not involving tetraethyl lead additives. Their pricing shows that a diesel converter for 400 cubic inch displacement engine would sell for approximately \$600. Gasoline engine converters for 200-300 cubic inch displacement are priced in the range \$250-\$350. Our sponsors wished to market similar products but at substantially lower prices. This need for cost reduction necessitated a literature review of all exhaust gas oxidizing systems, with emphasis on locating lower cost catalysts.

The most extensive work published in this field was by Ford Motor Company

engineers, Cannon and Welling, 2/ and Van Derveer and Chandler 3/. These men addressed themselves to the automotive exhaust gas problem with the associated problem of lead additive poisoning of the catalyst. Among the things they learned were: (1) platinum is quickly poisoned by lead additives 2/. (2) Copper oxide and chromium oxide mixed catalyst is very effective but easily poisoned by lead 2/. (3) Vanadium pentoxide is effective with hydrocarbons but not with carbon monoxide 2/. (4) At least 20% excess air is needed to sustain the reaction 3/. (5) Temperatures of 250-300° C. are needed to initiate reactions with the most effective catalysts 3/. (6) Among catalyst supports, α -alumina is considered best because of its high specific area and temperature stability 3/.

Confirmation of much of the Ford Company work was reported by S. Souriraja and Accomazzo 4/ who determined that CuO on α -alumina oxidized carbon monoxide (C) to 100% conversion above 200° C. Feenan, Swan, and Hofer 5/ demonstrated that 4% chromium oxide and 2% cupric oxide mixed catalyst gave very good oxidation of hydrocarbons and carbon monoxide at moderate temperatures of 200-300° C.

The only other inexpensive catalyst as effective as chromium or copper was cobalt. This metal was mentioned in many of the articles, but cobalt reacts with the alumina substrate to form an inactive cobalt aluminate 6/. The reaction can be inhibited by prereacting the alumina with calcium oxide but this reduces the specific area and doubles the cost of preparation 6/. Virtually no literature background was available on oxidation of diesel exhaust and our work in this area was an extension of the findings in automotive converter systems. A recent news item was noted 7/ which mentioned current developments by Universal Oil Products, Inc. of diesel exhaust converters.

III. EXPERIMENTAL WORK

A. Materials

1. Catalyst supports

The catalyst support chosen was Alcoa F-110, a catalyst grade alumina, 1/4" diameter spheres with an approximate surface area of 250 square meters per gram. This support is an optimum size to secure minimum feasible pressure drop with maximum surface area in an 0.25 to 1.0 cubic foot catalyst bed operating at 10,000 ft³/ft³/hr hourly space velocity 3/.

A limited amount of work was done with Alcoa H-151, 1/8" diameter spheres.

2. Catalysts

The following catalysts were used:

- (a) Platinum, 0.01% Pt
- (b) Chromium oxide with cupric oxide, 4% Cr-2% Cu
- (c) Cobalt oxide, 8% Co

These catalysts are designed to oxidize both hydrocarbon residues and carbon monoxide.

All reagents and materials, costs, and sources are listed in Appendix B.

B. Method of Preparation

The general method of catalyst preparation was to impregnate the support with the catalyst in the form of a water-soluble salt solution. The recommended amounts of metallic salts needed to form the catalysts as oxides on the support were computed. Then the salt was dissolved in a volume of water that would yield nearly complete absorption by the dry alumina. When the absorption was complete, the spheres were dried in an oven at 110° C. to a moisture content of about 0.1%. Then the spheres were calcined at 800° C.

for at least 1 hour, or until all fumes were evolved.

Procedural details and formulations for catalyst preparation are given in Appendix C.

C. Laboratory Testing

1. Apparatus and procedures

a. Micro-Converter

To test various catalyst systems, a micro-converter was set up using a 10 x 200 mm Vycor tube reactor into which was placed a preheating bed of uncoated alumina spheres, 1/8" diameter, followed by a 20 gram charge of test catalyst. Thermocouples were buried in the catalyst bed and suspended in the exhaust gas immediately downstream.

The fuel gas input to the converter was a single-component hydrocarbon from a gas scrubber bottle filled 2/3 full. Trimethyl pentane was used as a low boiling hydrocarbon, and benzene was used as a medium boiling hydrocarbon. A constant stream of air was bubbled through the hydrocarbon to effect complete saturation, then a controlled excess of air was added, and this total input was applied to the reactor, which was heated by an adjustable temperature tube furnace.

b. Temperature monitoring in the micro-converter

Temperature changes in the catalyst bed were monitored with a chromel-alumel thermocouple placed in intimate contact with the catalyst material, and similar thermocouples were used immediately downstream from the bed in the center of the gas stream to determine gas temperatures. Temperature-time records were taken with a recording pyrometer, and temperature inflection points were distinctly seen during linear programming of the power

input to the tube furnace. The initial inflection points of temperature versus time were used to define minimum activation temperatures for each catalyst as well as for different classes of hydrocarbons on any one catalyst. Figure 1 illustrates the data for the CrO₃/CuO catalyst system during a run.

C. Infrared analysis

The output of the micro-converter is sampled by a constant volume pump which delivers a stream of gas at 300 cc/min into a 10 cm path length infrared gas cell with 0.5 mil poly-propylene windows. The window absorptions are cancelled in the reference beam of a Perkin Elmer Model 700 double beam spectrophotometer. The output of the reactor is monitored over a wavelength range of 2.5 to 16 μ which includes hydrocarbon bands for alkanes, alkenes, alkynes, CO, CO₂, and H₂O, and most aromatics. By making an infrared spectrum while the reactor is cold one may establish a 0% conversion reference spectrum of absorption peaks. The final spectrum of known complete combustion at very high temperature establishes a 100% conversion spectrum. Then conversion of input products and formation of output products may be followed by the use of Beers and Lambert absorption laws:

$$A = \log \frac{1}{T} = a b c$$

Where: A = Absorbance

a = absorptivity

Percent Conversion =

T = Transmission
fraction

b = path length

$$\frac{C_{\text{orig}} - C_{\text{Final}}}{C_{\text{orig}}} \times 100$$

c = concentration

Thus one may determine percent conversion for different temperatures or hourly space velocities. Figure 2 shows infrared data as taken with a chromium oxide - copper oxide catalyst system. Figure 3 shows the derived plots of absorbance versus percent conversion of hydrocarbon, and Figure 4 shows percent conversion of hydrocarbon versus temperature for this catalyst system.

d. Chromatographic analysis

Gas-liquid vapor chromatography was also used to study the inputs and outputs of both the micro-reactor in the laboratory and full size prototype converters in the field. The typical sampling technique utilized a 3 to 5 cc gas-tight syringe of sample injected into a Perkin Elmer Vapor Fractometer Model 154D, thermal conductivity detector.

The columns generally used were a 3 ft. by 1/8" ID gel column for CO and CO₂ and O₂ analysis and a 6 ft. by 1/8" ID polypropylene glycol on Chromosorb P column for CO₂ and hydrocarbon residues. Column temperatures were room temperature to 70° C. for most analyses with approximately 80 cc/min of helium flow. Adequate resolution was sought on CO, CO₂, and O₂, with only approximate resolution sought for hydrocarbons. For hydrocarbon analysis only, an integrated output was taken from an electronic printing integrator attached to the gas chromatograph.

2. Laboratory Results

Laboratory test results are summarized in Table I. One observes that all catalyst systems studied yield substantial conversion of hydrocarbons and carbon monoxide. Best performance was attained with the Cr-Cu system, and fortuitously, that is also the most economical catalyst.

Additional micro-converter work with leaded gasoline demonstrated the susceptibility of platinum catalyst to lead poisoning, and the resistance of the cobalt-passivated catalyst to this effect. Unfortunately, the latter system yields relatively poor conversions. The lead poisoning susceptibility of the Cr-Cu was not determined, because at this point in the project, work was oriented exclusively toward the diesel converter problem. Diesel fuel

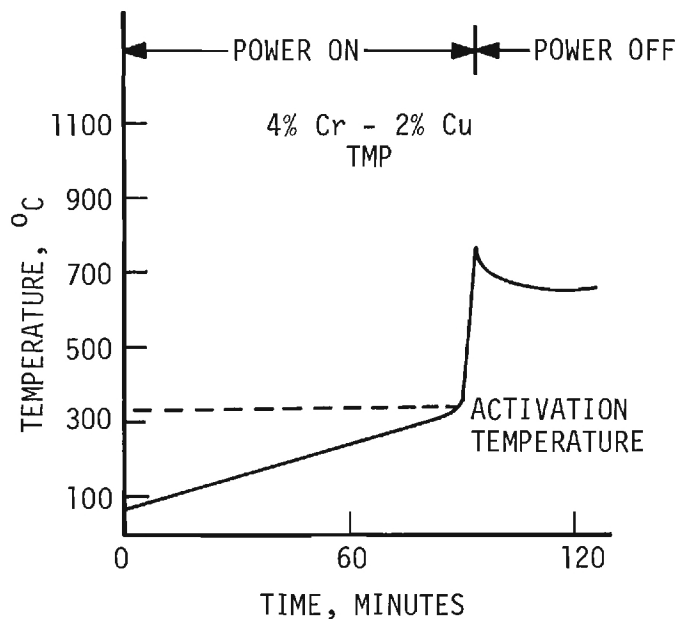


Figure 1. Recorder plot showing activation temperature.

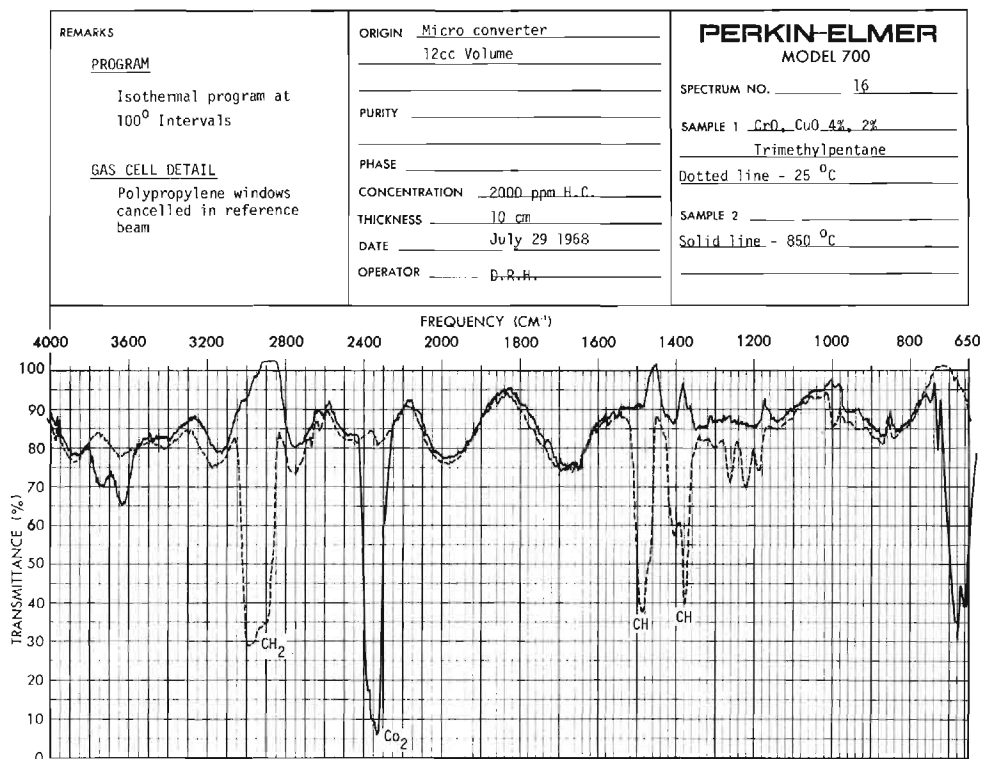


Figure 2. Infrared spectrum of exhaust gases from Cr-Cu converter

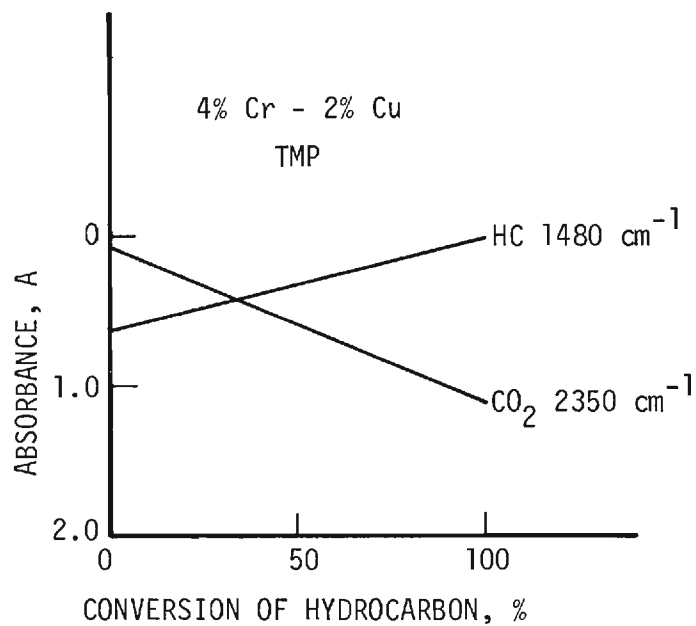


Figure 3. Percent conversion of Hydrocarbon vs. absorbance.

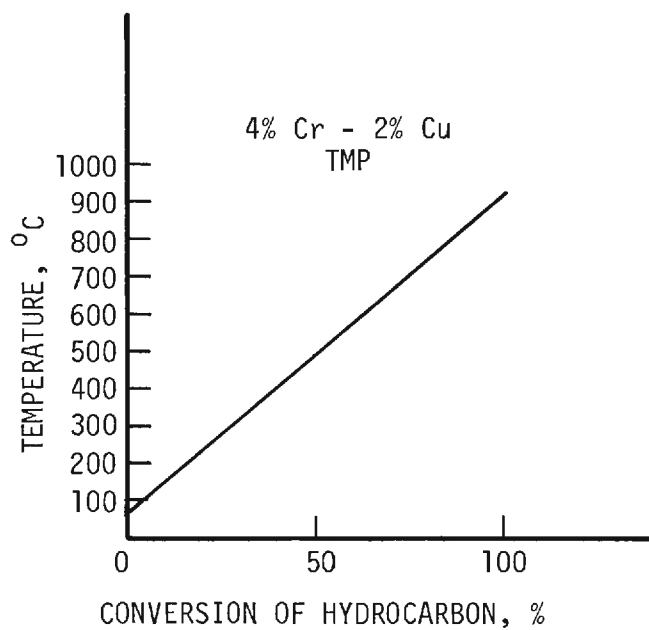


Figure 4. Percent conversion of Hydrocarbon vs. temperature.

TABLE I
MICRO-CONVERTER TEST RESULTS

Catalyst System	Activation Temp., °C			Conversions, % at Indicated Temperatures, °C									
	TMP	Benzene	CO	TMP				Benzene				Co	
				°C	%	°C	%	°C	%	°C	%	°C	%
Cr, Ce, Fe, Th	300	--	300	--	--	--	--	--	--	--	--	500°	100
0.01% Pt	300	--	--	450°	99	850°	99	400°	50	850°	100	450°	99
8% Co, CaAl ₂ O ₄	350	275	--	450°	10	850°	50	600°	18	850°	80	--	--
4% Cr 2% Cu	350	350	--	450°	48	850°	90-99	450°	40	850°	87	850	99

does not contain the leaded anti-knock additives that are responsible for the poisoning effects from gasoline fuels.

D. Operational testing

Full scale prototype converters for both automotive exhausts and diesel exhausts were designed and fabricated. Figure 5 is a drawing of the basic design. Each converter was to be tested for carbon monoxide conversion, back pressure characteristics, hydrocarbon cleanup, odor reduction and smoke reduction. Table II is a summary of the converters built, and of all available test results. The bulk of the diesel prototypes were completed by September 15, 1968 and delivered to Mr. L. R. Daugherty, for placement at various field sites. Subsequent tests on these converters were planned, however, only a small part of the testing was accomplished prior to termination of this project. The companies who received the converters for testing have been very pleased with an obvious reduction in odor, smoke, and noise. It has been noted that the prototypes reduce diesel engine noise substantially better than conventional mufflers.

1. Tests at National Air Pollution Control Center, Virginia Avenue, Cincinnati, Ohio

0.01% platinum and 8% cobalt oxide converters were taken to the air pollution control laboratories (APCL) on Virginia Avenue, Cincinnati, Ohio, July 10, 1968, with the intention of running them without excess air as muffler replacements for passenger vehicles. The tests were run on 1963 Ford with a Clayton Dynamometer using the California Cycle 8/ as a test. None of the devices yielded better than 20% conversion of either hydrocarbons or carbon monoxide. The maximum temperatures that were reached were only

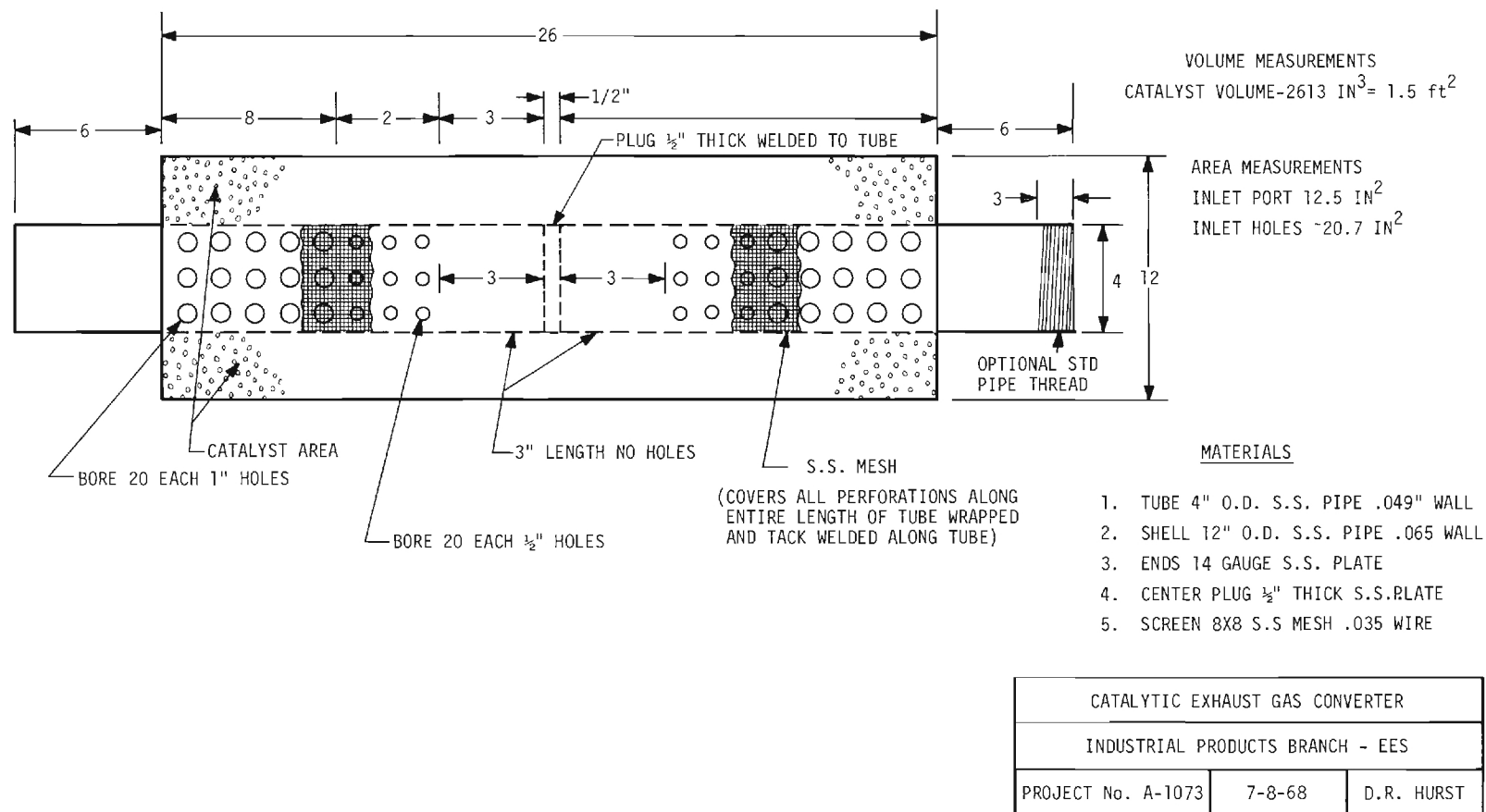


Figure 5. Drawing of Diesel prototype converter.

TABLE II
Evaluations of Prototype Converters

Engine Type	Converter Description						Test Conditions				Performance Data			
	Size		*Shell Material	Catalyst Support		Catalyst	Test Site	Engine Displacement--in ³	Excess Air -%	Back Pressure @ Max. Load	Conversion % HC CO	Odor	Smoke	Eye Irritants
	Dia. In.	Length In.		Type	Size	Metal Oxides % of Metal								
Gasoline	6	32	MS	Mesh	--	Cr,Ce,Fe,Th	E.E.S.	300	None	--	None	--	--	--
	4x8	18	MS	Mesh	--	Cr,Ce,Fe,Th	E.E.S.	300	None	--	None	--	--	--
	8	32	MS	Mesh	--	Cr,Ce,Fe,Th	E.E.S.	300	None	--	None	--	--	--
	4x8	18	SS	F-110	1/4"	0.01%Pt	APCL	285	None	--	20 20	--	--	--
	6	18	MS	H-151	1/8"	0.01%Pt	APCL	285	None	excessive	20 20	--	--	--
	6	18	MS	F-110	1/4"	8%Co, CaAl ₂ O ₄	APCL	285	None	--	None	--	--	--
	6	18	MS	H-151	1/8"	8%Co, CaAl ₂ O ₄	APCL	285	None	excessive	None	--	--	--
Diesel	12	28	SS	F-110	1/4"	0.01%Pt.	Blalock Eng.	400	~150	3 - 4" Hg	-- 99%	good	good	good
	12	26	SS	F-110	1/4"	4%Cr 2%Cu	Manitowoc	400	~150	--	-- --	good	good	good
	12	26	SS	F-110	1/4"	4%Cr 2%Cu	Grayhound	600	~150	--	-- 99%	good	excell- ent	good
	12	26	SS	F-110	1/4"	4%Cr 2%Cu	Knoxville	400	~150	--	-- --	--	--	--
	8	26	SS	F-110	1/4"	4%Cr 2%Cu	Univ. of Tenn.	--	--	--	-- --	--	--	--
	8	26	SS	F-110	1/4"	4%Cr 2%Cu	Chem. Prod. Mines	600	~150	--	-- --	good	good	good
	8	26	SS	F-110	1/4"	4%Cr 2%Cu	---	--	--	--	-- --	--	--	--
	8	26	SS	F-110	1/4"	4%Cr 2%Cu	---	--	--	--	-- --	--	--	--

*MS = Mild Steel
SS = Stainless Steel

275-300° C., and it was obvious that there was insufficient air and insufficient heat available. A considerable amount of literature was obtained on this trip and a review of this literature indicated that at least 20% excess air would be needed for gasoline powered engines. Temperature requirements were also discussed with a minimum requirement of 350° C. being indicated for almost all converters tested.

2. Blalock

A 28 inch long, 12 inch in diameter converter was filled with platinum .01% on α -alumina 1/4" diameter spheres and taken to Blalock Machinery and Equipment Company, Inc., 225 Forsyth Street, S. W., Atlanta, Georgia for testing on a GMC 6-Cylinder 400 cubic inch diesel engine mounted on a test block and capable of being loaded to 200 hp. at 1850 rpm. Initial tests with a MSA carbon monoxide tester indicated 99% clean up of 0.5% CO level in the exhaust. A hydrocarbon test was attempted* but the results were inconclusive.

3. Greyhound

Further tests were run on September 27, 1968 at the Greyhound Maintenance Dept., 5260 Peachtree Industrial Blvd., Chamblee, Georgia. There a 12 inch diameter 26 inch long converter with Chromium-Cupric catalyst was placed on a 671 cubic inch disp. diesel commonly used in the large bus industry. Carbon monoxide tests were run with the results of 99% clean up of CO after the converter with approximately 0.01% input to the converter at 1900 rpm, no load.

*Samples of 30 cc of exhaust gas taken before and after the converter by trapping in carbon tetrachloride (CCl_4) were examined in an infrared spectrophotometer. The absence of absorption differences at 2985cm^{-1} as compared with pure CCl_4 reagent suggest that the sample size was insufficient.

4. Manitowoc

Two converters using chromium oxide and cupric oxide on α -alumina were built and placed on large diesel engines used on a local crane erector stationed in Atlanta. Quantitative data from these tests was not available at the time of project termination, but should be included in future work.

5. Other tests

Two chromium oxide, cupric oxide diesel converters were taken via Mr. Daugherty to test sites in Tennessee. One was placed on a Knoxville Transit Authority bus, the other was sent to Mr. Barney Fish at the University of Tennessee. No reports were available from these test sites as of this writing.

One similar diesel converter was placed in service at a mining company at Cartersville, Georgia. Mr. Daugherty reported that clean up of smoke, odor, and eye irritants was very good. Substantial heating within the converter was noted, under heavy load.

IV. DISCUSSION OF RESULTS

The predominant emphasis of this project work has been towards achieving workable converter units, and demonstrating their utility and economy with an absolute minimum of technical effort.

Accordingly, extensive experimentation to explore catalyst systems, and define optimum materials and processing conditions was not undertaken. Maximum use has been made of the existing technical literature with rational adaptation to the problem at hand. We believe that the results indicate that the approach was satisfactory to the extent that a suitable catalytic converter system was developed for practical use on diesel engines. Further, the

experimental work has identified the factors responsible for special difficulties in developing gasoline engine converters. Specifically, the special problems of catalyst poisoning by leaded gasoline, control of excess air to the converter, and maintenance of operating temperatures have been recognized as requiring extensive technical study. For this reason, further exploratory work on gasoline converters was deferred in favor of more complete development of the diesel units.

Clearly, the laboratory work has been technically sound to the extent that practical diesel converters have been produced. At the same time, we recognize that some of the laboratory methods will require further development or modification to provide reliable production quality control and to yield more precise measurements for future developmental work.

The manufacturing costs of the developed converters may only be approximated at this time, but the use of available material costs may be assumed to provide a conservative estimate. The following specific data is applicable to a converter designed for a 400 in³ diesel engine:

Stainless steel converter body, fabricated ----- \$100.00
Catalyst Charge:

<u>Material</u>	<u>Weight</u>	<u>Cost/lb.</u>	
Alumina Spheres	50	0.35	\$17.50
Chromic Anhydride	4	1.73	6.94
Cupric Nitrate	4	1.34	5.35
			<u>\$29.79</u>
Catalyst Manufacture	53	0.10	<u>5.30</u>
			TOTAL ----- \$135.09

If the price to the user were twice the cost figure, then the above unit should sell for \$270. With conventional mufflers for such units priced in the neighborhood of \$100, this price does not appear to be at a level that

would discourage demand. By crediting the conventional muffler cost to the converter unit, a cost of \$170 for the pollution abatement would seem quite tolerable.

In summary, the experimental work has led to a technically and economically satisfactory catalytic converter unit for diesel engines -- the mechanical design of the unit, and the basic process for producing the supported catalyst.

V. CONCLUSIONS

1. An economically feasible catalytic converter system has been developed for use on diesel engines.

2. The catalytic systems studies have the basic capability for converting gasoline engine exhausts, but substantial technical problems have not been solved.

3. More comprehensive technical studies of the catalyst composition, processing variables, equipment and economics are needed as a basis for final decisions on manufacture and marketing of a diesel exhaust converter.

4. Extensive field data on performance of diesel converter units in actual service is needed as a basis for advertising literature, marketing and service programs, and for feedback to the developmental work.

VI. RECOMMENDATIONS

1. This investigation has been addressed entirely to the technical aspects of exhaust converter development. We now recommend commercial development of diesel converter systems as feasible.

2. We have reviewed the published and patent literature and believe that all materials used in this development work have been cited in the open

literature. We cannot, however, preclude the possible existence of infringed design, process, or composition of matter patents. Accordingly, we recommend that an infringement patent search be undertaken based on the disclosures in this report to determine the status of this development both with respect to existing active patents and possible patentability of any aspects of this development.

3. Since current technical news reports have indicated that others are working on diesel converters, and since air pollution legislation could create sudden large demands, preparations for commercialization should be accelerated. Accordingly, we recommend that pilot-scale technical development work be undertaken to establish design and process control parameters for manufacturing operations and to fully evaluate the economics, performance and servicing requirements of diesel exhaust converters. A proposal covering these activities is being prepared.

VII. APPENDIX

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A. Bibliography

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B. Materials and Sources

<u>Materials</u>	<u>Cost/lb.</u>	<u>Source</u>
α -Alumina		
Type F-110 1/4" Sphere	0.35	Aluminum Company of America, Inc.
Type H-151 1/8" Sphere		
Chromic Anhydride, C. P.	1.73	J. T. Baker Chemical Company
Cupric Nitrate, Tech.	1.34	J. T. Baker Chemical Company
Calcium Nitrate, Tech.	0.15	J. T. Baker Chemical Company
Cobalt Nitrate, Purified	0.86	J. T. Baker Chemical Company
Platinum Chloride, C. P.	2040.00 (based on Pt)	International Nickel Corp.

C. Catalyst Preparation

Catalyst materials were processed in the laboratory in accordance with the following procedures:

Platinum

Dilute a 10.0 gm. solution of 10% Pt as the platinic chloride to 2 liters of water and then add 10,000 gm or approximately 22 pounds of 1/4" α -alumina spheres. Soak the spheres with agitation until all is absorbed, then dry and calcine at 700° C. for two hours.

Cobalt Oxide on Passivated Alumina

Take 10% by weight of calcium nitrate based on weight of alumina spheres and dissolve in 25% by weight of water, based on weight of alumina spheres. Soak spheres in this solution until all is absorbed. Dry the spheres in an oven at 110° C. then calcine at 700° C. for 2 hours or until all nitrogen oxide fumes are evolved.

Pour warm spheres into a solution made of 32% (by weight of alumina spheres) cobaltous nitrate dissolved in (25% by weight of water based on alumina spheres). Soak the spheres until all of the solution is absorbed then dry at 100° C. and calcine at 700° C. The catalyst is then ready for use.

Chromium Oxide Cupric Oxide

Make a solution of 8% chromium anhydride based on alumina spheres and 8% cupric nitrate based on alumina spheres by dissolving these chemicals in 25% by weight of water based on the alumina weight. Soak the spheres with agitation until all the solution is absorbed, then dry at 110° C. and calcine at 700° C. for 2 hours or until all nitrogen oxide fumes have evolved.

D. FORMULATIONS

The foregoing procedures (C. Catalyst Preparation) correspond to the following materials formulations:

<u>Materials</u>	<u>Solutions to treat 100 lbs. of Alumina Spheres</u>			
	Platinum	Ca	Co	Cr-Cu
	<u>Passivation</u> (quantities in lbs.)			
Platinum-Chloride Sol'n, 10% Pt	0.1	--	--	--
Cobaltous Nitrate	--	--	32	--
Calcium Nitrate	--	10	--	--
Chromic Anhydride	--	--	--	8
Cupric Nitrate	--	--	--	8
Water	20	25	25	25