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PROJECT N“: Brite EuRarn Proposal BE-6146

TITLE: Nitrogen oxide removal from diesel and lean otto engine exhaust
LeaNOx

PROJECT
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PROGRAMME

DATE: 12.03,1996

Project Description

The object of the LeaNOx project was to research catalytic materials and processes for the conversion of nitrogen oxides in net axidising exhaust-gas atmosphere. The familiar 3-way catalytic converter technology cannot be used for this condition, occurring for example in Diesel engines and lean-burn spark-ignition engines. Since lean-burn engines have exceptional future potential due to their favorable consumption properties, the lack of an effective NOX aftertreatment technology presents a hurdle which must be overcome if minimisation of consumption and other improvements in emissions are to be attuned to each other.

The first specific work on catalytic nitrogen oxide conversion by means of hydrocarbons under high oxygen excess was started at about the same time, at the end of the 1980s, in Europe and Japan (1-4). A literature and patent search accompanying the LeaNOx project has to date revealed over 300 sources, underlining the high level of strategic and scientific interest in the subject.

The LeaNOx project was carried out by an international consortium from a total of six European countries, with the financial support of the European Community. The members of the consortium were the vehicle manufacturers shown in Figure 1, together with research institutes in the fields of materiats/catalysis and engine technology. The project was supported by a total of six catalyst manufacturers, who themselves were not integrated into the project, but were associated with the project by a co-operation agreement.

The project comprised

- Basic research into the elementary stages of lean NOX catalysis.

- Formulation and experimental pre-selection of catalyst samples from various groups of catalytically active materials

- Application-oriented screening of samples of various cata[yst manufacturers

- Activity tests on catalytic converters and system testing on a lean-burn spark-ignition engine, a swirl-chamber Diesel engine and a direct-injection Diesel engine in a car, and a direct-injection Diesel engine for a truck.’

Laboratory Results

The tests were carried out with a variety of reducing agents, including laboratory gas compounds with a mixture of gaseous hydrocarbons, with petrol and Diesel, and in a few random sampling tests with an aqueous urea solution.

Figure 2 shows an overview of the catalytic materials investigated at the various university research institutes, as well as the respective maximums of the nitrogen oxide conversion rates obtained under laboratory conditions and the catalyst temperature at which this conversion was achieved. [t was generally seen that a whole range of solid body structures can selectively reduce nitrogen oxides with hydrocarbons in a temperature range >350° C, but conversion rates of> 20% with a complete exhaust gas, including water vapour, were achieved only when noble metals were deployed to increase activity. In this case the solid body structures served only as substrates for the noble metal, and determined its fine distribution. The addition of zinc to platinum catalysts on aluminium oxide brought about a reduction in N_zO formation under laboratory conditions.

Figure 3 and 4 give two examples of the catalytic activity of solid body structures, partly with a noble metal coating. The SCR-activity of perovskites, $AB0_3$ has been investigated in several meanings

a) activity of pure perovskites

b) promotion of activity by impregnation with noble metals

c) activities of composite materials with perovskites and solid acids.

The composites have been found to show the best catalytic behaviour. For dry synthetic exhaust gas with 2000 ppm NO, 1330 ppm C#6 / C_3H_8 (2: 1 mix), 2 !4 0_2 and lie as balance, activity data of three different $BiFe0_3$/ $Ti0_2$ composites have been summarized in Figure 3. The composites 1 ?4 $BiFe0_3$/ $Ti0_2$ ahd 3 ?40 $BiFe0_3$/ $Ti0_2$ show the best conversion of NO into N_2 with a selectivity towards N_2-production >0,8 in the entire temperature range. However, the addition of water vapour to the synthetic exhaust inhibited the NO-conversion strongly.

The system $La_2Zr_20_7$ had been investigated, using different calcination temperatures and different noble metal loadings between O ‘A Pt and 2 ‘A Pt, The best result was obtained with the catalyst 1 % Pt/$La_2Zr_20_7$, calcined at 550° C. As is shown in Figure 4, this catalyst gives up to 40 % conversion into N_2 at a test gas composition of 500 ppm N(3, 6 Yo 0_2, 220 ppm C_3H_8, 110 ppm CSH6, 350 ppm CO, 12 % H_20, fl O % $C0_2$. The activity was maintained during repeated activity tests, however, a catalyst aging at a temperature of 700° C led to a considerable deterioration of the activity.

A special role is played by zeolites, into which transition metais are introduced. Figure 5 shows how various transition metal ions affect the conversion properties of zeolites for nitrogen oxides. In addition to the familiar and well-documented copper-based catalysts (5, 6), a metal combination was found which produced particularly high levels of nitrogen oxide conversion and a particularly broad working temperature range under the }aboratory conditions.

All catalytic materials examined under laboratory conditions at the universities were in the form of powders or granulates, This meant that translation of the results to monolithic catalytic converters used in practice was not directly possible because, at identical space velocity, the catalyst mass available for the conversion of a specific mass flow of nitrogen oxides is substantially higher for a powder-form oatalyst than when the powder is applied to a monolith.

Figure 6 shows, as an example, the attempt to translate the test results with the best catalytic material from the screening of the various zecdite catalysts onto a monolith. [f the same catalyst mass associated with a space velocity of 40.000 h-~ in the powder experiment is applied to a monolith and the gas flow rate would remain constant, a space veiocity of only 3.000 h-~ would result. If the flow rate through this monolith is then increased to 30.000 h-l, a significantly lower catalyst activity results. This modelling result was verified by an appropriate preparation of a monolith with the relevant catalytic material. The conclusion is that experiments under laboratory conditions with powder- or granulate-form catalysts can only be compared among themselves, and that translation into quantitative resu{ts on monolithic catalytic converters in the laboratory, or even on monolithic catalytic converters in practical engine use, is not possible.

Screening of Monolithic Catalyst Substrates

The application-oriented screening with catalyst samples in monolith form on the catalyst test rig was carried out principally with catalysts from the companies associated by co-operation agreement with the LeaNOx project. Catalysts from other sources, procured worldwide, were however investigated for comparison purposes

A typical result with a noble-metal catalyst is shown in Figure 7. The test gas composition on which the consortium had agreed, designated EUCAR test gas, is shown in Figure 8. The catalyst reveals properties which essentially enable it to be applied in the low-temperature range; that is to say, in Diesel exhaust-gas for exampte. It shows a maximum nitrogen oxide reduction of approx. 28°A at a temperature of 200” C, and has a working temperature range with nitrogen oxide conversion ~ 10% between 170° C and 270” C. Some 2/3 of the reduced nitrogen oxide becomes nitrogen, the rest N_PO. In addition to the selective nitrogen oxide reduction, a considerable NO_z formation is observed in this catalytic ponverter.

The second group investigated in addition to these highly-active low-temperature catalysts were catalyst materials with an optimum working temperature range around 350° C. These cata[ytic materials were mostly zeolite-based, with transition metals as active components. A useful combination which can cover a broad range of working temperatures **is a** high-temperature catalytic converter positioned at the front in direction of flow with a low-temperature converter arranged behind it. The most favorable combination examined on the test rig is shown in Figure 9. With it, nitrogen oxide conversion rates of up to 40% can be achieved under the given conditions. The formation of by-products, that is to say production of N_20 and NO_z, is considerably more favorable in the high-temperature catalysts than in the low-temperature ones. The actual conditions at the fitting location under the given conditions during engine/vehicle running are the deciding factors in practical engine application. The catalytic **converter combination must be matched to those conditions.**

Urea as a reducing agent aHows for substantially higher levels of NC3X conversion, as shown in Figure 10. Both VzO#TiOz catalysts in widespread use in SCR systems with ammonia, as well as zeolites, are suitable in principle as catalysts. [n addition to the criteria of maximum NOX conversion and temperature window, which in the example shown in the illustration favours the V@J170$_2$catalysts, other criteria such as dynamic response, formation of by-products and NH~ Jeakage are decisive in practical application. At least for cars with fast-running Diesel engines in passenger cars, there is still a lack of comprehensive system observation in this area. The basic problem is of course the logistic disadvantage of carrying an additional reducing agent in a separate tank.

Engine Testing Results

Results from a lean burn gasoline engine running on a test bench are shown in Figure 11 for a CU-ZSM5 catalyst and Figure 12 for a structured type where the zeolite is followed by a Pt catalyst. The engine has 4 valveslcylinder, a displacement of 21 and was tuned by some modifications for lean operation up to k = 1.5. NOX and hydrocarbons in the exhaust gas could be varied within a wide range by altering airlfuel ratio, ignition and injection timing. Therefore it was possible to vary the HCI IN OX ratios between 2 and 30. Temperature of the exhaust gas was kept constant by a closed loop cooling system. An additional injection of any reducing agent was not necessary, because it was proved that the hydrocarbons from the combustion process are quite appropriate reductants. With regard to a constant NOx-reduction this fact may lead on the other hand to problems as far as the composition of hydrocarbons in the exhaust gas is strongiy depending on the blend of aromatics, olefines and paraffined in the fuei. This parameter is implicitly contained in Figure 11.

Figure 1 f shows the conversion rates of nitrogen oxides and hydrocarbons for a typical high-temperature catalyst at engine operating points corresponding to a space velocity of 30.000 h-l cm the catalytic converter. in this, the lower margins of the NOX spread are associated with a I-ICINOX ratio of 2, and the upper margins of the spread apply to a HCINOX ratio of 10. In the latter case conversion rates of up to 60% are achievable. This conversion rate would correlate with an extremely lean engine setting, the untreated nitrogen oxide emission values being correspondingly low and the HC values already correspondingly high. As an overall effect of tow untreated emission and high catalytic conversion a very favorable level of NOX emission would thereby be achievable downstream of the catalytic converter, although at the cost of increased HC emissions.

This HC problem and the level of nitrogen oxide conversion at low temperatures can be improved by a combination of noble metal and zeolite catalysts. A combination of this kind is shown in Figure 12 and indicates that, with this option, from temperatures of approximately 230° C, conversion rates of around 20°A can be achieved. These then rise to approximately 60°/0 in the main operating range of a catal~ic converter of this kind of a lean-burn spark-ignition engine. Due to the lower volume of the high-temperature catalytic converter in this case, however, the operating range for high nitrogen oxide conversions is shifted to higher temperatures and is also narrower than if the entire volume were to consist of the high-temperature catalyst.

A fundamental restriction of the use of Cu-zeo[ite catalysts is a rapid and severe ageing process when treated with reducing exhaust gas. The reason for this is the change of oxidation state of the copper ion introduced into the zeolite necessary for selective nitrogen oxide conversion. If this catalyst is exposed to reducing exhaust-gas conditions, the copper R reduced down to the metal and the catalyst will thus lose its effect. A catalytic converter combination as per Figure 10 could consequently be used only for a genuine lean-bum engine, but not for a lean mix engine. Therefore, for the lean portion of the operating performance map of lean mix engines, noble metal catalysts must be deve[oped with an appropriate temperature range of catalytic activity. Catalysts of this kind were not yet available to the LeahK)x project, however.

Further possibilities for exhaust-gas aftertreatment of lean mix engines are the so-called NOX storing catalysts (7 - 9). This type of catalysts was only researched at a basic level in the LeaNOx project, to investigate the individual stages of catalytic conversion, but not in practical applications.

Figure 13 shows a characterizing result of the research into nitrogen oxide conversion on a swirl-chamber Diesel engine. The entries made are conversion rates resulting for a platinum NOx catalyst, when additional fuel is injected into the Diesel exhaust gas upstream of the catalytic converter, producing a hydrocarbons to nitrogen oxides ratio of 4 I-ICIANO. The results obtained can be correlated effectively with the catalyst input temperature as the basic parameter. The engine speed, and thus the space velocity, were of lesser importance for this catalyst in the parameter range tested. In the temperature range around 200° C the catalytic converter achieves a maximum conversion rate of some **305%0.** At exhaust-gas temperatures ~ 300° C, that is to say in the higher load ranges, nitrogen oxide reductions are no longer achieved. An extrapolation of the nitrogen conversion to be expected with this catalytic converter in the MVEG test produced an expected mean NOx conversion rate of 10 %. Since this estimate was not able to incorporate the non-stationary behaviour of the catalytic converter, it does of course entail a degree of uncertainty.

The results shown in Figure 13 were achieved with a fresh catalyst. Ageing of various catalysts on the engine test rig revealed that a deterioration in catalytic activity is not necessarily to be expected as a consequence, as shown in Figure 14. of the four catalytic converters tested in the ageing programme$_{d}$ the catalyst containing transition metals and one of the three noble metal catalysts showed negative ageing effects. However, the behaviour of the other noble metal cataiysts improved slightly in the course of ageing. The reason for this is likely to be that, due to the ageing, the catalyst reduced its very high activity for the oxidation of hydrocarbons with oxygen, so that a higher proportion of hydrocarbons remained available for selective nitrogen oxide reduction. At the same time, the ageing caused the optimum NOX conversion window in this catalyst to shift to higher temperatures.

Further measurements of selective nitrogen oxide conversion in car Diesel engines were carried out on a 1.9 I DI Diesel engine with common rail injection. Figure 15 shows, by means of the reducing agent ratio parameter, the NOX and iiC emissions upstream and downstream of the catalytic converter and the resuiting efficiency of the catalytic converter. The I-f C/NOx ratio was adjusted in this case by additional post

injections by means of the common rail system. It is seen that, with the catalytic converter used, NOX conversion rates of up to 40% can be achieved at high liC-to-NOx ratios. Increased breakthrough of hydrocarbons is also observed at the same time, however, due in part to the increased untreated HC emissions and, secondly, also to overloading and consequent reduction in efficiency of the catalytic converter. A HUNOX ratio of >5 appears to be unrealistic as a possibility when seen in these terms. Observation of the additional fuel requirement also leads to a similar result. An additional injection, bringing about a tiC/NOx ratio of 5 in the exhaust gas, already increases fuel consumption by 4%.

Whereas the first-generation noble metal catalyst shown in Figure 15 requires comparatively high H(YNOX ratios for nitrogen oxide conversion, with the second-generation noble metal catalyst measurable nitrogen oxide conversion rates are possible even with the hydrocarbon mixture present in the exhaust gas without additional injection. Figure 16 shows results recorded on a 1.300 kg weight class vehicle with a 1.9 I DI Diesel engine with common rail injection. It is seen that the catalytic converter used here reduces the nitrogen oxide emissions by some 18°A even without additional fuel injection. At the same time, the HC and CO emissions are reduced by 60 to 70Y0. The introduction of additional reducing agents by way of the common rail system reduced the nitrogen oxide emissions by a further $3%, although this caused the HC emission to rise significantly again, and the CO emission to rise slightly. The additional fuel consumption was around 3Y0. Even if the nitrogen oxide conversion rates with this system are still far removed from the conversion rates to which we are nowadays accustomed with 3-way catalytic converters, these catalysts do represent an important aid to compliance with the more stringent future emission limits.

The testing of nitrogen oxide conversion on commercial vehicle engines was carried out on a turbocharged 7 16-cylinder IX Diesel engine with intercooling. Figure 17 shows the NOX conversion rates for various catalysts at two selected stationary operating points, representing test points in the R49 test. The resulting weighted mean values for the overall R49 test are also shown. However, no equipment was certificated and the engine parameters and the hydrocarbon injection were changed manually and not simultaneously. It is seen that, while low-temperature catalyst achieve comparatively good conversion rates in the low-load ranges, their conversion rate at higher loads fails to such an extent that conversion rates of c 10% are to be expected in the overall R49 test. Through a combination of high-temperature and low-temperature catalysts the overall conversion rate tin be raised to approximately 35% at a HC_1}NOx ratio of 4, or 44% at a HC_1/NOx ratio of 6. The additional fuel consumption was determined 4,2 % for HC_1/NOx = 4 and to 6 % at I-fClINOx = 6.

Consequently here, too, it is seen that the deployment of latest-generation catalysts enables conversion rates which indicate that lean nitrogen oxide conversion with hydrocarbons as a reducing agent is a real option for the future.

Conclusions and Prospects

- Methods of catalytic conversion of nitrogen oxide in lean exhaust gas are on the threshold of usability. However, their efficiency is still far removed from that of the 3-way catalytic converter, and it is extremely questionable whether comparable efficiencies will ever be reached.

- Highly active noble-metal catalysts such as are required for the low exhaust-gas temperature level of Diesel cars permit a maximum of 25!40 NOX conversion in the operating range described by the MVEG test. Formation of undesirable byproducts, like N@ and N0$_2$ is an additional criterion for the further development of these cata&st types.

- High- and low-temperature catalyst combinaticms allow conversion rates up to approximately **40%** in truck engines in the R49 test, and 20 to 25940 in lean-burn spark-ignition engines in the MVEG test.

- For engines which have to be run under stoichiometric or rich conditions for periods of time (lean mix engines), only noble metal-based lean catalysts are usable, because zeolite catalysts with transition metals are not resistant to ageing. Exhaust hydrocarbons from lean gasoline engines are well suited as reducing agents. Additional injection of reducing agent is not necessary.

- Research in this field must be intensified in order to be able to achieve the objectives of reduced consumption and C02 reduction with simultaneous reduction of nitrogen oxide emissions.

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Relationship Between the LeaNOx Partners

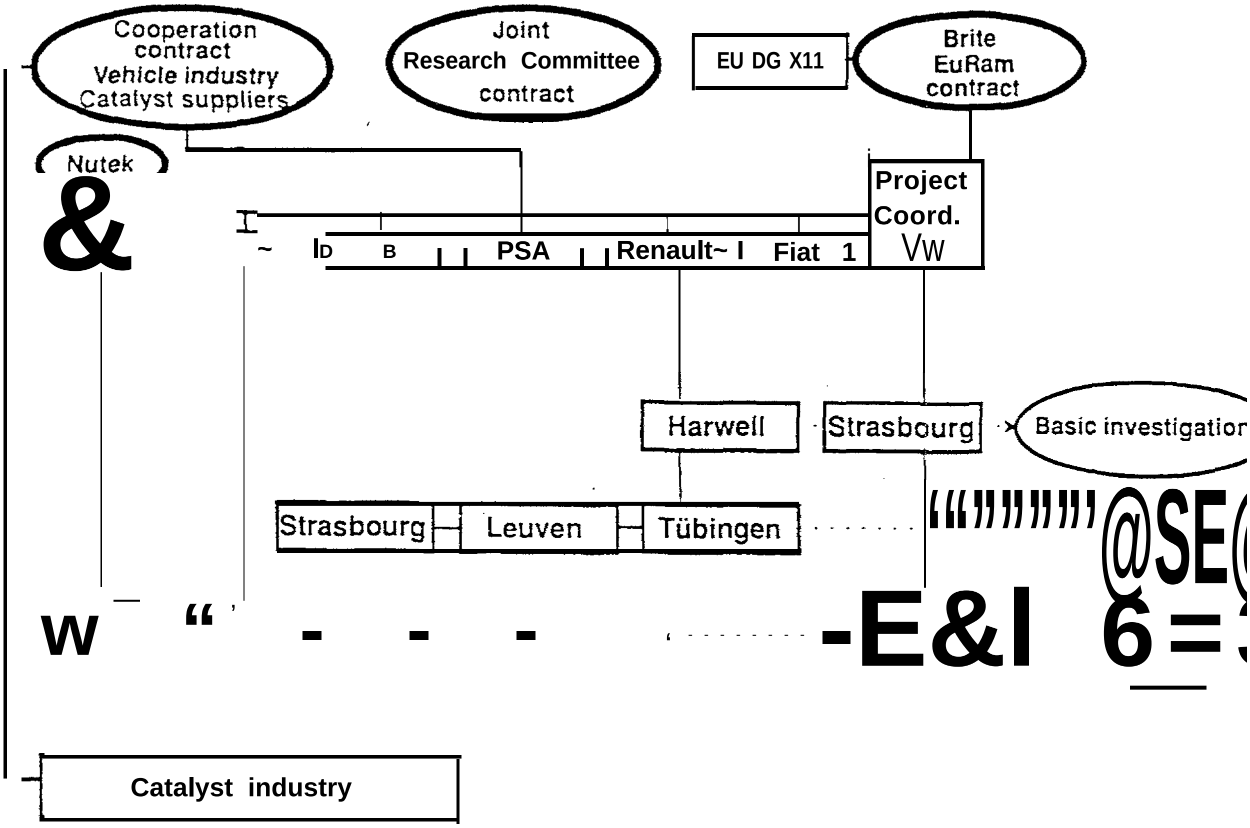

Figure 1

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Catalyst Systems Investigated

- Platinum, Pal[adium/Copper, Zinc - Systems
  No significant improvements of conversion rate compared to conventional noble metai catal

- Perowskites (e. g. Bi(Fe,Rh)O,, and (M, Cu,hln)Fe,,,Mn,O, with M = Ca, La)
  Convers@n rate too low and range of operating temperature too high (<21 “A above 3504 C

- Solid acids of composition Flh or BiFeO, coated on TiO, or ZrO,
  Conversion rate on BiFeO, 30-34 o/., with F?h 45-56 o/.

- Mixed oxides of Cu/Ce
  Conversion behaviour unsatisfactory, two maxima, 19% at 230 “ C and 16 ‘/~ at 400 “ C

- Pyrochlores (A,$B_2$0, with A = La, Sm and B = Zr, Ti) with/without Pt - impregnation
  Activity is depending on the crystal structure of the surface, interesting only with Platinum impregnation (42 % at 280 ‘ C}

- Zeolites (Cu-, Co-, Fe-, Ni-, Ag-, Ga-exchanged)
  Good results on powder catalysts (up to 80 ‘/c. conversion), not yet reproducible on monolith samples

Table SCR-activity and surface area of 13iFe03/Ti02-composites

	max. N2 production conversion ‘A	at (“C)	surface area (~2fg)
15 % BiFe0$_3$/Ti0$_2$	24	350	82
3 ?40 BiFe0$_3$1Ti0$_2$	35	300	79
1 Yo BiFeOaiTi07	34	300	76

Figure 3

Activity of 1 ?-4 Pt/La2Zr207 cakined at 5500 C

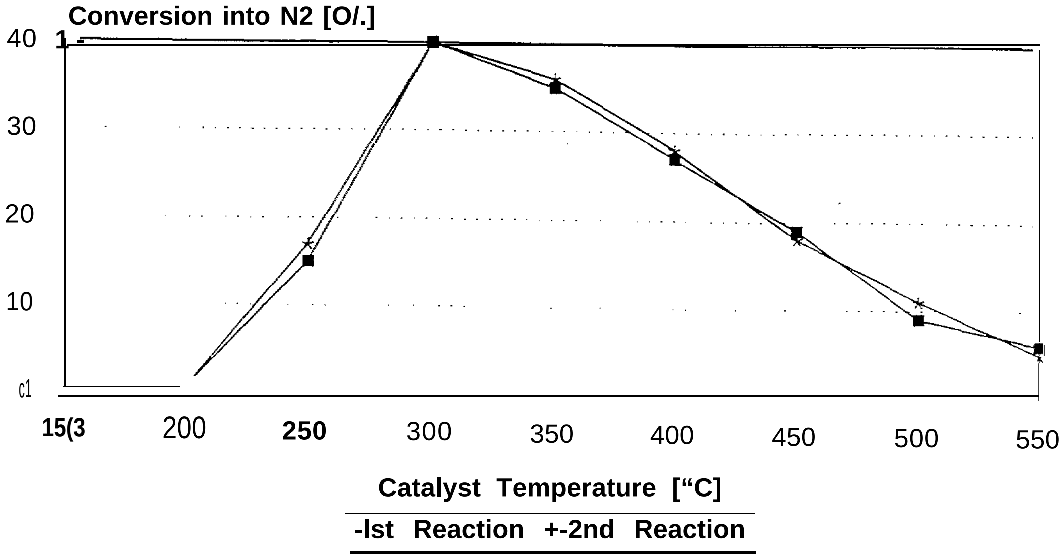
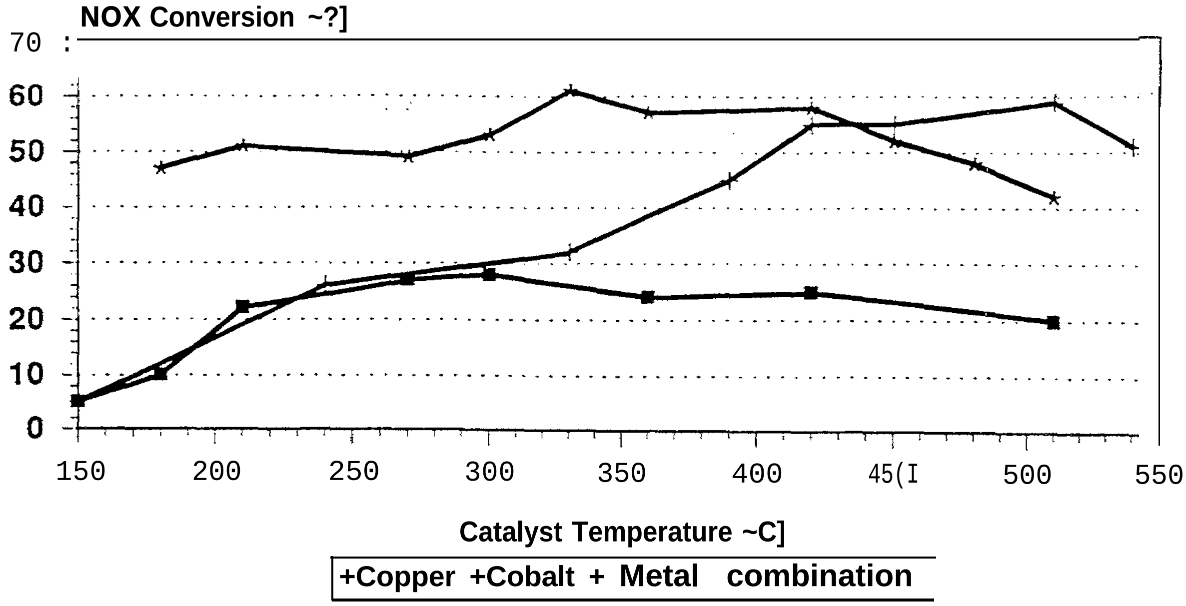


Figure 4

Transition Metal Catalyst on Beta-Zeolite

HC : NO= 2, Space velocity 20.000/h



12% H2C), 10 ‘A C02, 6 % 02,350 ppm CO, 500 ppm NO, 350 ppm Propane/Propene (1 :1), Helium balance

Figure 5

Effect of Space Velocity on NOX Conversion

Metal combination catalyst, powcferform and coated on monolith

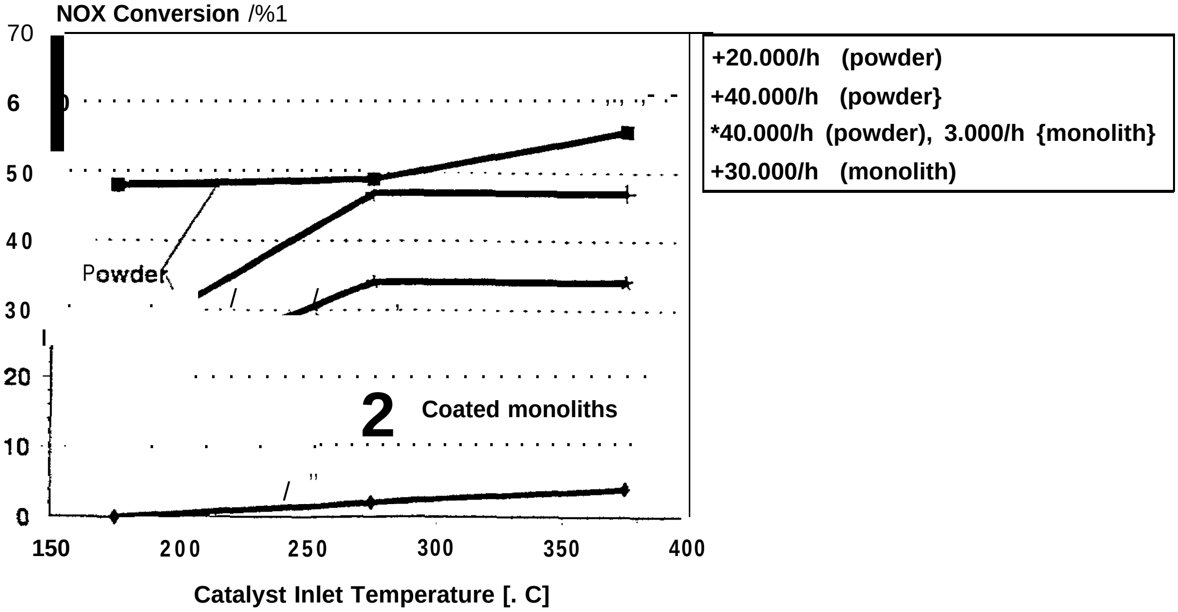


Figure 6

Piatirwm Catalyst m Cmdierite (50 g/cft)

ELICAR **lestgas** c~ntainfng **Kerosene (IIU :NO=4), Space Velocfty** W,wum

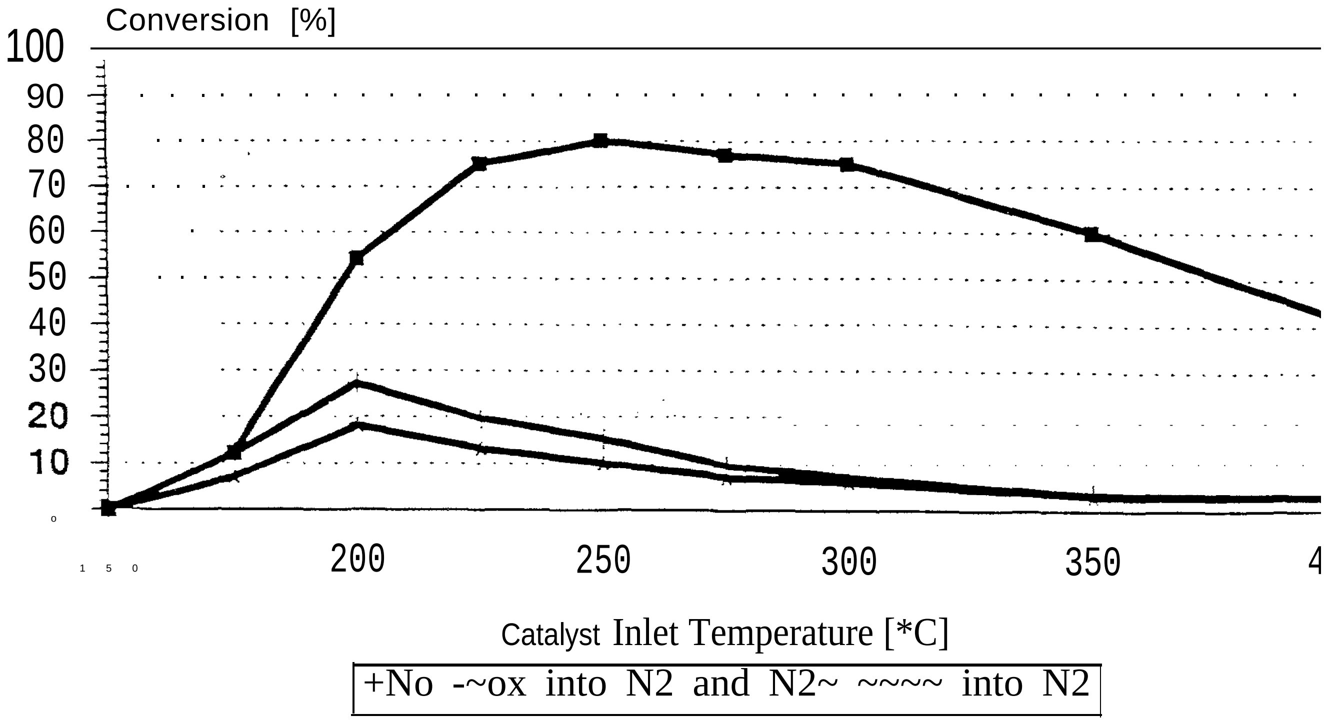


Figure 7

Catalyst Test Conditions

Gas composition:

O_2	6 ?40
co	350 ppm
co,	1070
t-l,0	1270
No	50(3 ppm (HC, : W =4)
Hc	660 ppm HC, (Propan/Propen 2:1 Gasoline or Kerosene)
N,	Balance

Using carbamide instead of HC the

n~~H,},CO / n_{NO} ratio is adjusted to 0,5

Space velocity: 30.000 h-’

Temperature range: 150-550*C ‘

Catalyst Combination (CWt) an Cordierite

I-K : NO = 4, **Space velocity 30.000/h**

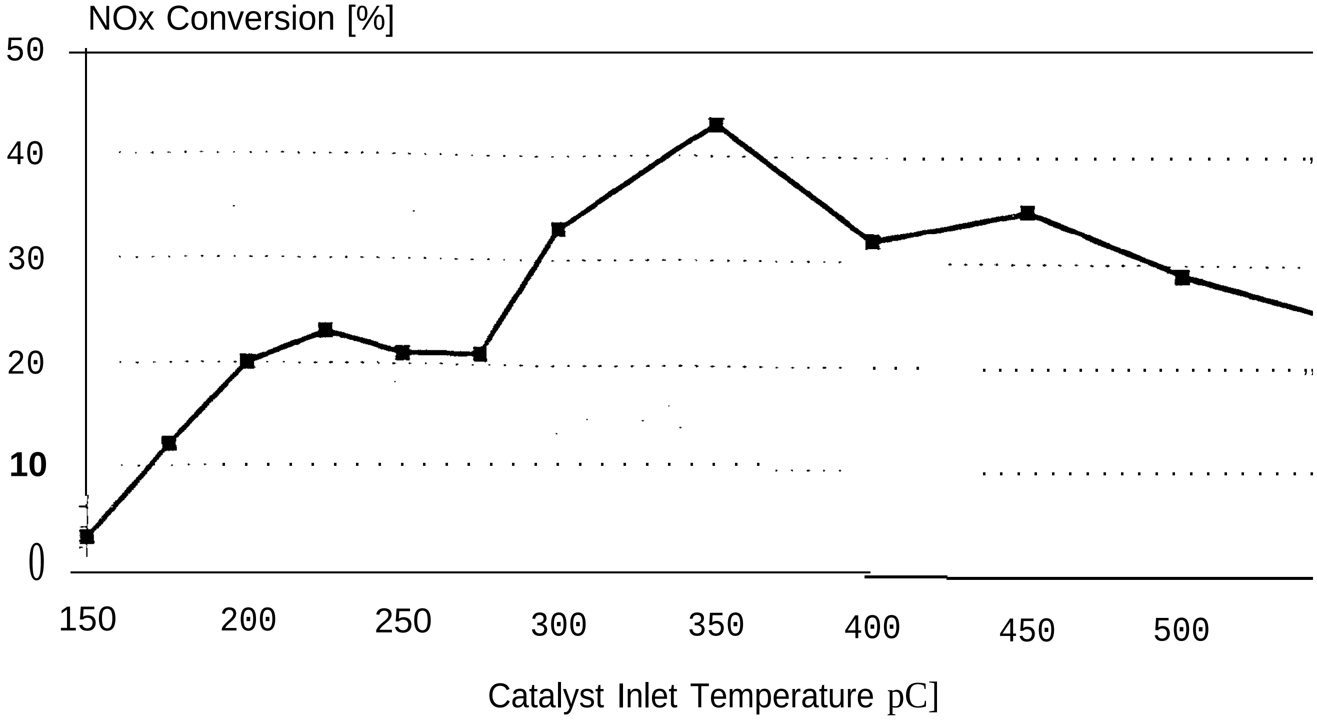
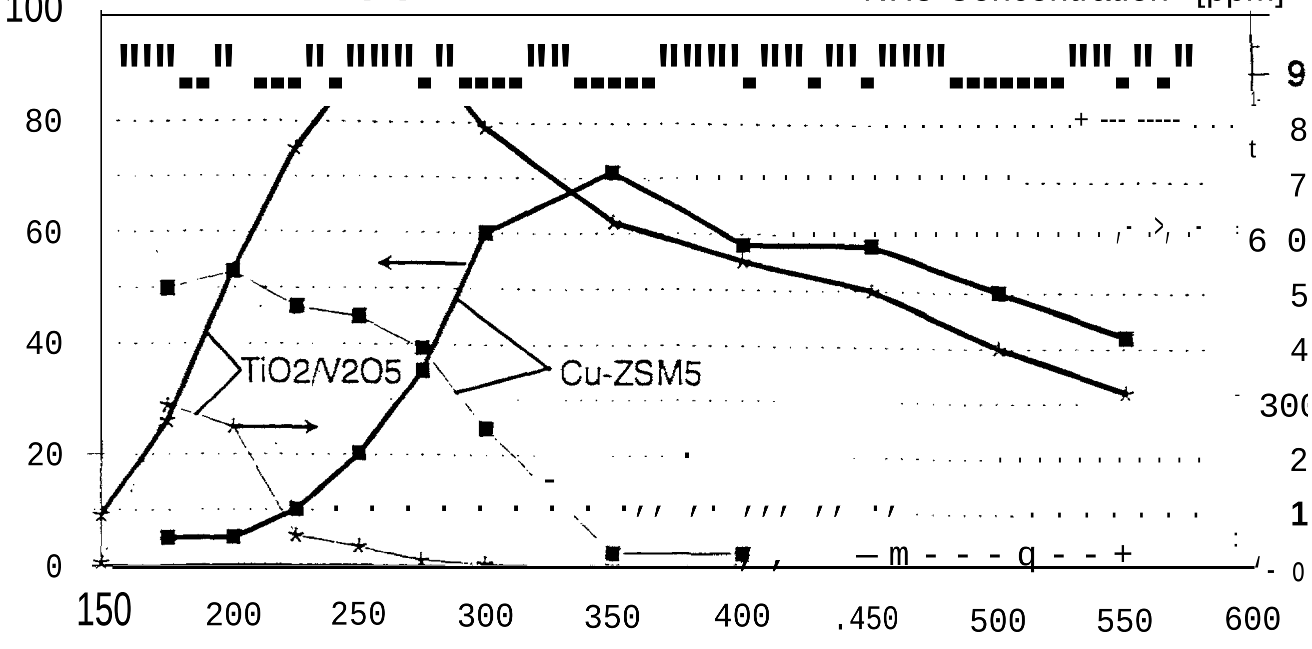


Figure 9

NOX Reduction Using Carbamide

Space velocity 30.000/h, **Stoichiornetric ratio 1,00**



Emission Reduction on Lean Burn Gasoline Engine

Industrial CU-ZSFA5 catalyst, space velocity 30.000/h

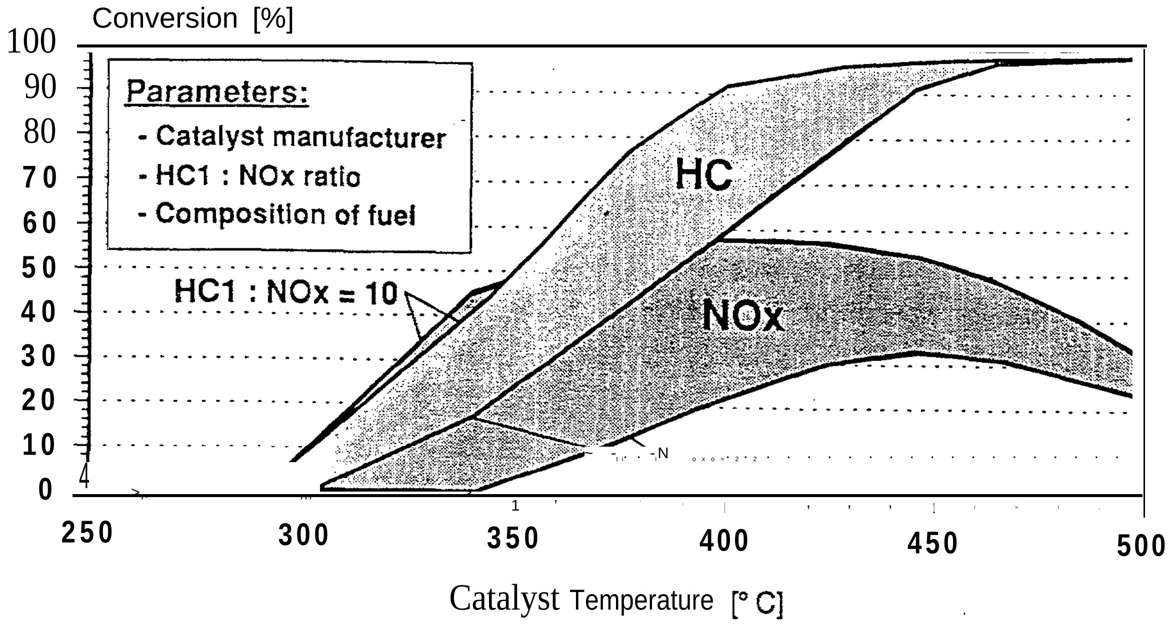


Figure 11

Emission Reduction on Lean Burn Gasoline Engine

Pt/Cu-ZSM5 catalyst, space velocity 30.000/h, HC1 : NOX = 2-10

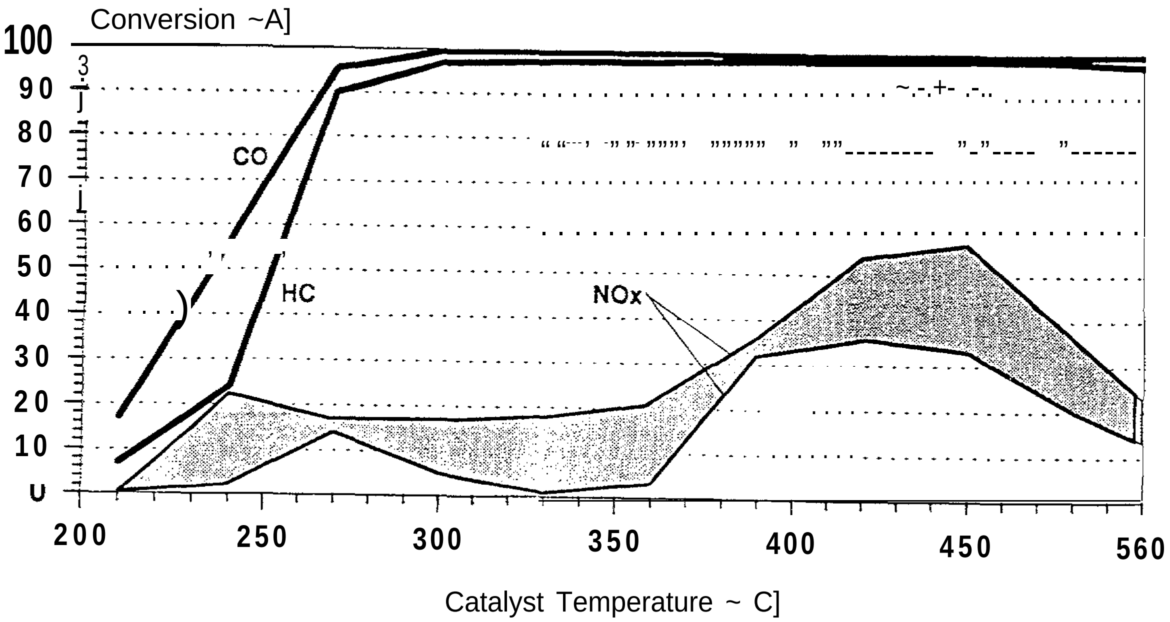


Figure 12

Platinum Catalyst Investigation on Diesel Engine

Reducing agent: Diesel **fuel (HC1 :NO=8)**

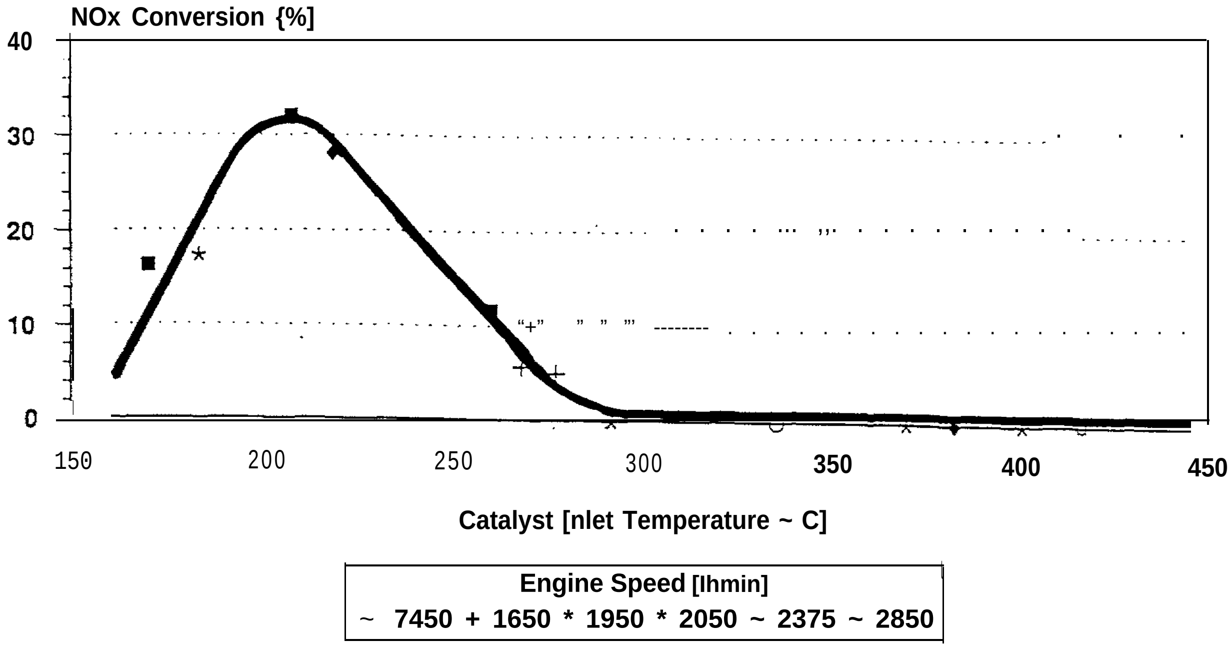
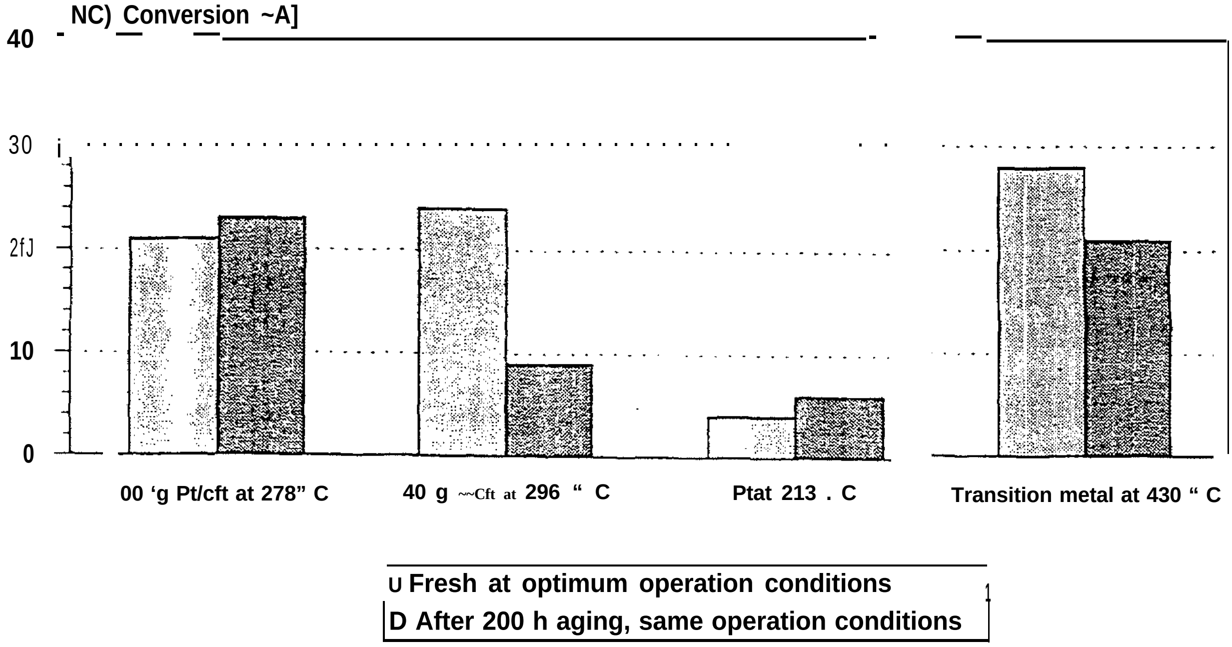


Figure 13

Catalyst Aging on Diesel Engine

Experiments with Kerosene under steady state conditions, HCI : NO= 8



Aging cycle: 0.5 h at 350 " C, 0.5 h at 500 " C, 1.0 h cooling down at engine stop

Figure 14

Effect of tiC/NOx Ratio on Emission and Increase of Fuel Consum

1.9 I Diesel engine equipped with common rail and noble metal catalyst

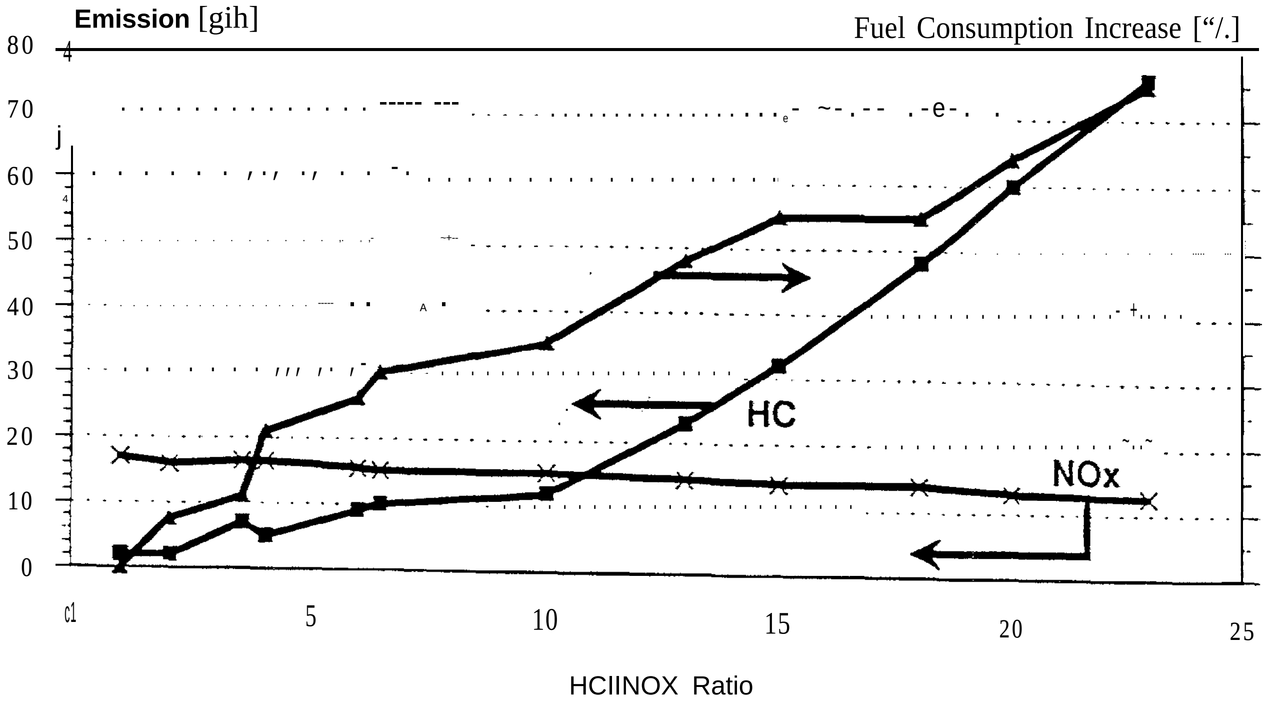


Figure 15

ECE/EUDC Roller Bench Tests

1.S! 1 D1-Diesel Engine with cooled EGF3 and kiw temperature catalyst

	HC [g/km]	CO [g/km]	No$_x$ [g/km]	CO, [glkm]
Raw emission (w/o catalyst)	0,20	1,4	0,51	175
With catalyst (w/o reducing agent)	0,07	0,42	0,42	175
With catalyst (with reducing agent)	0,13	0,45	0,39	180

Heavy Duty Truck D1-Diesel Engine

Turbocharged 7 I - Six cylinder DI Diesel Engine with intercooler

	Steady state conditions		ECE R 49 Test procedure
Engine speed [rnin’] Percent of max. torque [Y.] pm, [bar] Catalyst inlet temperature {“C]	1400 100 13,0 485	2400 10 1,0 237	
Low temperature catalyst Iic,mdor = 4	570	28 “h	8,%
High/low temperature catalyst combination HC,/’NO, = 4	42 “/o	9 0/0	35 ‘!0
-iigh/low temperature catalyst combination -f C,/t@3, = 6	527.	13%	44 ‘?’lo

Figure 17