The Effect of Fuel and Oil Additives on Automobile Catalyst Performance

THE SUITABILITY OF PLATINUM METALS CONFIRMED

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Stringent standards controlling the emission of exhaust pollutants from gasoline-fuelled spark ignition engines have necessitated studies of many factors that influence the efficiency of the catalysts used for the destruction of carbon monoxide, unburnt hydrocarbons and nitrogen oxides. This review of the literature considers the effects on the catalyst of lead, manganese and phosphorus, which may be present as additives in fuel or lubricating oils. The first two elements can be detrimental to catalytic activity, and also to the environment, so must be minimised. The deactivation caused by phosphorus can be avoided by the use of catalysts designed to be compatable with the specified lubricants. Catalysts containing platinum group metals remain the preferred means of controlling the three designated exhaust gases.

Since automobile catalysts have come onto the market, a considerable amount of work has been carried out on the effects of fuel and oil contaminants on catalyst activity. A large number of papers have been published on the subject, particularly with respect to lead and phosphorus.

In the early 1970s, studies concentrated on oxidation catalysts, but more recently the effects on three-way catalysts have predominated in the literature. This article will review some of these findings and will reference only studies relating to monolithic-supported three-way catalysts published from 1976 onwards.

All the pollution control catalysts considered here contain platinum group metals, specifically combinations of platinum and rhodium. It is well known that these metals not only exhibit good low temperature performance and high temperature stability in an exhaust environment, but are also more poison resistant than base metal catalysts.

Emission legislation currently in force in the U.S.A. requires catalysts to be durable for a minimum of 50,000 road miles. In California it is intended that by 1993 the emissions from most vehicles will be reduced still further, see Table I, with a requirement for 100,000 miles durability (already applicable for light duty trucks). More recently, proposals have been tabled by Congressmen Waxman and Dingell to halve the 1993 values.

European Legislation

In the European Community, legislation has been passed which requires a lowering of the emissions from vehicles of less than 1.4 litre engine capacity to levels considered equivalent to current U.S. standards, which are given in Table II. It is intended that Directives will be passed in 1990 to ensure that by the end of 1992 all vehicles, regardless of engine capacity, will meet these lower limits; specifically 19 g/test carbon monoxide and 5 g/test hydrocarbons plus nitrogen oxides. It is also intended that a higher speed extra urban driving cycle will be included to follow on directly from the Economic Commission for Europe urban cycle. The emission limits will reflect a

Table I
United States Emission Standards

Model year	Pollutant, grams per mile		
	Carbon monoxide	Hydrocarbons	Nitrogen oxides
Federal			
1975	15	1.5	3.1
1980	7	0.41	2.0
1983	3.4	0.41	1.0
California			
1977	9	0.41	1.5
1983	7	0.41	0.7
1993	3.4	0.25	0.4

combination of these two cycles, but the proposed values are still under discussion. Also under discussion is the introduction of a 80,000 km durability cycle similar to that used in the U.S.A.

It is clear therefore, that by the end of 1992 three-way catalysts will be fitted to all new cars in the European Economic Community (E.E.C.). More protective emission standards and extended durability requirements mean that contamination by poisons and their effects on catalyst activity will be important factors in the overall life of the emission system. The review will therefore examine the effects of lead and manganese in the fuel, and phosphorus and heavy metals in the oil. The results of misfuelling studies in the U.S.A. will also be considered.

Catalyst Deactivation By Lead
Lead Content of Petrol

Over the last decade many researchers have studied the effects of European leaded petrol, typically containing 0.4 or 0.15 g/l lead on the performance of both oxidation and three-way catalyst formulations, some of which were designed to be lead tolerant. Now, however, the E.E.C. has enacted legislation requiring unleaded petrol to be marketed within the Community. By the same legislation all new

Table II
European Community Emission Standards

Engine size	Dates of introduction		Pollutants, grams per test		
	New models	All new cars	Carbon monoxide	Hydrocarbons + nitrogen oxides	Nitrogen oxides
Over 2 litres	1/10/88	1/10/89	25	6.5	3.5
1.4–2 litres	1/10/91	1/10/93	30	8	—
Under 1.4 litres					
Stage 1	1/10/90	1/10/91	45	15	6
Stage 2	1/07/92	31/12/92	19	5	—

Table III

Effect of Lead Levels in Fuel on the Activity of Pulsator-Aged Catalysts[a]

Fuel[b], mg lead per litre	Simulated miles × 1000	Conversion, per cent									Hydrocarbon light off temperature for 80 per cent conversion
		Pulsator (at 500°C) R[c] = 1.15			Steady-state (at 550°C)						
					R[c] = 1.05			R[c] = 1.60			
		NO_x	CO	HC	Net NO_x	CO	HC	Gross/net NO_x	CO	HC	
3	14.4	67	67	63	98	98	95	94/82	41	66	373°C
5	15.5	47	61	50	96	98	96	96/88	48	52	425°C
10	15.0	22	33	37	92	95	92	68/67	45	52	—

[a] Platinum/rhodium three-way converter (5:1 at 38 g/ft³) aged on European ageing cycle [b] Isooctane fuel including 0.2 mg phosphorus/l and 0.03 wt.% sulphur [c] R = ratio of reductants to oxidants

cars sold from October 1989 must be designed to run on unleaded petrol.

It has been shown that catalyst deactivation by lead is dependent upon the amount of lead deposited on the catalyst, and whereas this is likely to increase with higher lead contents in petrol, many other factors must also be taken into account, including catalyst formulation, catalyst ageing, operating conditions, and interactive effects of lead with other fuel and engine oil additives.

Most of the research work undertaken on this topic has involved the use of either laboratory combustion rigs or test bench engines for catalyst ageing, and synthetic exhaust gas rigs for catalyst performance evaluation (1, 2, 3). There is no standard universal test procedure for laboratory studies, and in view of the effects of varying conditions including gas temperature and air:fuel ratio, it is perhaps not surprising that reported data are varied and in some cases conflicting. More recently, work has been carried out with vehicles under actual road driving conditions (4).

The durability of three-way catalysts for European Community applications has been examined by investigating their activity as a function of the higher temperatures encountered in autobahn driving modes over extended periods of time (1). Lead levels of 1, 3, 5 and 10 mg/l were evaluated in pulsator durability and dynamometer ageing studies, Table III. Platinum-rhodium catalyst loadings of 40 g/ft³, and in the ratio of 5:1, maintained substantial three-way conversions, when aged under rich conditions at maximum temperatures of 900–1000°C with 3 mg/l lead. Ageing at these temperatures resulted in sufficient reduction of BET surface area, to cause increased stoichiometric hydrocarbon light-off temperatures. Net nitrogen oxides and hydrocarbon conversions after light-off were improved due to lower lead retention on the catalyst. Increasing residual lead levels to 10 mg/l, when ageing in pulse flame reactors at 1000°C, much decreased three-way catalyst performance after 4000 simulated miles.

The effects of lead levels of 3 and 10 mg/l on

Table IV

Summary of Emission Results for the 80,000 km Durability Vehicles

Vehicle number	Distance, km × 10³	Emissions, grams per mile			Conversion, per cent			Remarks
		HC	CO	NOx	HC	CO	NOx	
		0.32	2.26	0.77				Legal level assuming 1.3 Deterioration Factor
1	0	0.285	2.24	0.26	86.4	80.9	91.3	
	6.5	0.509	4.32	0.38	79.9	67.7	89.3	
	50	1.012	7.66	0.41	71.4	55.6	86.3	Aged Hego[a] 10 mg Pb/l
	80	1.260	6.83	0.56	68.6	54.1	81.8	Aged Hego
	80	0.748	4.14	0.87	75.1	66.3	73.8	Fresh Hego
2	0	0.248	1.07	0.61	89.4	89.3	80.7	
	6.5	0.418	2.52	0.58	83.8	77.5	84.6	Trace Pb
	50	0.479	3.92	0.45	83.03	71.93	84.8	Aged Hego
3	6.5	0.152	1.36	0.62	89.2	88.7	85.4	
	80	0.607	6.00	0.70	65.0	69.4	83.6	Aged Hego 10 mg Pb/l
	80	0.358	2.86	1.03	76.7	79.1	76.2	Fresh Hego
4	0	0.156	1.01	0.26	90.5	89.6	88.6	10 mg Pb/l
	6.5	0.358	2.24	0.63	78.3	82.0	84.2	
	50	0.675	3.32	1.18	71.8	69.0	59.7	Aged Hego
5	0	0.175	0.85	0.44	88.8	—	—	
	6.5	0.184	1.16	0.70	89.8	86.9	77.0	Trace Pb
	50	0.216	1.47	1.37	90.0	80.4	52.5	Aged Hego

[a]Hego = heated exhaust gas oxygen sensor

three-way catalyst activity were examined after ageing in pulsator, dynamometer and vehicle trials (4). This work showed that after pulsator ageing at maximum temperatures of 1000°C, catalysts aged with 10 mg/l lead were more deactivated than those aged with 3 mg/l, the effect being more noticeable when the air:fuel composition was rich of stoichiometry, and under perturbation conditions. Reducing the maximum ageing temperature to 730°C caused a greater reduction in activity than at 1000°C for a catalyst aged with 3 mg/l lead, even though the surface area of the catalyst aged at the lower temperature was 50 per cent higher. It was concluded that the poisoning effect of lead deposited at the lower temperature was more significant to catalyst efficiency than the loss of this amount of surface area.

Dynamometer ageing to the equivalent of 80,000 km Automotive Manufacturers Associa-tion (A.M.A.) driving cycle, followed by vehicle testing, showed that with 3 mg/l lead the vehicle met U.S. legislated limits, but with 10 mg/l lead only nitrogen oxides were within the legal limit, while hydrocarbon and carbon monoxide emissions were significantly above. A number of vehicles were tested between 50,000 and 80,000 km A.M.A. road cycle driving, with either 10 mg/l lead or trace lead fuel. These tests showed quite clearly that with 10 mg/l lead it was not possible to meet U.S. legislation limits due in part to poisoning of the oxygen sensor. However, with trace lead fuel this was quite possible, see Table IV.

Final conclusions from this work were that lead levels of 3 mg/l should not cause concern under any driving conditions; but continuous long term exposure to levels of 10 mg/l are unacceptable for long term catalyst and oxygen sensor durability. The European Community

Table V
U.S. Motor Vehicle Manufacturers Association Fuel Surveys

Year	Unleaded regular gasoline	
	lead, mg per litre	lead, grams per U.S. gallon
1975	1.8	0.007
1976	2.1	0.008
1977	1.8	0.007
1978	1.8	0.007
1979	4.2	0.016
1980	2.9	0.011
1981	2.9	0.011
1982	1.3	0.005
1983	0.8	0.003
1984	0.8	0.003

1984 Leaded regular gasoline contains 213 mg Pb/l

standard for unleaded fuel allows a maximum lead level of 13 mg/l. However, market surveys show that in West Germany, Sweden, Switzerland and Austria levels of less than 2.5 mg/l are the norm. Similarly, in the United Kingdom, since unleaded fuel has become more freely available, lead levels measured in the field are considerably lower than the maximum and decreasing. Current U.S. and Japanese unleaded fuel contains less than 1 mg/l of lead, Table V. Similar trends would be expected in the Community as 1992 approaches.

Effects of Vehicle Misfuelling

In the past, deliberate misfuelling, that is the use of leaded petrol in vehicles designed to use unleaded fuel, has been a problem in the U.S.A. The main reasons for this occurrence were that leaded fuel was slightly cheaper than unleaded, and that some owners erroneously perceived improvements in vehicle performance when using leaded fuel. In the mid-1970s, during the fuel shortage, some owners were indiscriminate in their choice, and

sometimes unleaded fuel was unavailable at the more remote filling stations, resulting in the misfuelling of catalyst equipped vehicles.

In Europe, unleaded fuel is generally cheaper than leaded, and this removes any incentive to car owners to deliberately tamper with their vehicles in order to misfuel. However, the possibility will exist as long as a supply of leaded fuel for older cars is still available. Similar precautions have been taken in Europe as in the U.S.A. in that all cars designed to run on unleaded fuel are fitted with narrower orifices to the fuel tank than vehicles designed for leaded fuel. The petrol pumps dispensing unleaded fuel have correspondingly narrower filler nozzles. Despite these steps, U.S. experience has shown that misfuelling can occur, if the incentive of cheaper leaded fuel exists. Fuel pricing policies in Europe, where unleaded fuel is cheaper, should minimise this possibility.

Two studies conducted by the Environmental Protection Agency showed that the principal effects of misfuelling are increases in the emissions of all three regulated pollutants. The most detrimental effect is the increase in hydrocarbon emissions, followed, in order of severity, by carbon monoxide and nitrogen oxides (5, 6). The frequency of misfuelling is less important than the total quantity of leaded fuel consumed.
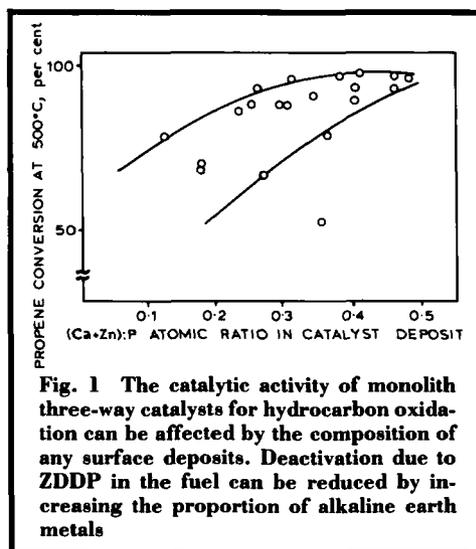

Fig. 1 The catalytic activity of monolith three-way catalysts for hydrocarbon oxidation can be affected by the composition of any surface deposits. Deactivation due to ZDDP in the fuel can be reduced by increasing the proportion of alkaline earth metals

Even if misfuelling is intermittent, it can eventually cause major catalyst deactivation. Some degree of reactivation of misfuelled catalysts is possible, provided the misfuelling incidents are small in number and intermittent, though reactivation is never complete; the hydrocarbon activity is the least recoverable.

It is evident that even minor incidents of misfuelling are detrimental to catalyst activity and the measures already taken to discourage and prevent misfuelling must be continued.

Catalyst Deactivation by Manganese

In North America, methylcyclopentadienyl manganese tricarbonyl (MMT), was introduced in 1974 as an alternative, or supplement, to lead anti-knock compounds in fuel. Although its use in unleaded fuel was disallowed in the U.S.A. in 1979, it is still added to leaded fuel in that country, and has been a constituent of 90 per cent of the unleaded fuel used in Canada for over ten years.

During combustion, most manganese is converted to the oxide hausmannite (Mn_3O_4), the formation of which is not affected by the absence or presence of other fuel additives (7). Hausmannite does not act as a chemical deactivator, but where catalyst inlet temperatures greater than 850°C are encountered it can build up on the catalyst substrate causing the mouth of the pores to be narrowed, and may eventually plug the monolith channels. (8). Both effects result in catalyst deactivation, and these deposits can only be removed from the catalyst by physical means.

The combustion of fuel containing MMT gives rise to higher unburnt hydrocarbon levels than MMT-free fuels, with consequent effects of higher hydrocarbon tailpipe emissions (7, 8, 9).

Data generated during the Coordinating Research Council's MMT Field Test Programme showed no occurrence of catalyst plugging (9). This sixty-three-car 50,000 mile durability programme was undertaken using maximum fuel MMT concentrations of 16 mg/l. A field test undertaken by Environment

Canada has shown that 1983–85 model year cars operating on unleaded petrol containing MMT will meet the 1988 Canadian emissions standard of 0.41 g/mile hydrocarbon.

More recently a study to determine the effect of MMT on performance was conducted on a series of in-use catalysts that were operated in Canada, and which had completed between 22,000 and 43,000 miles (10). The authors confirmed deposition of Mn_3O_4 on the catalyst surface, and at least some channel blockage. This was accompanied by a decrease in functional activity and an increase in light-off over the catalyst.

Catalyst Deactivation by Phosphorus

In this article only the effect of oil derived phosphorus on catalyst activity will be considered. Some laboratory reactor and test bench engine studies have shown that engine oil-derived phosphorus can cause both three-way and oxidation catalysts to lose activity (11, 12). The major source of phosphorus in exhaust gas is likely to be from zinc dialkyl dithio phosphate (ZDDP) present in lubricating oil which reaches the combustion chamber past the piston rings and valve stem seals. Both combusted and uncombusted forms of ZDDP may occur in exhaust gas, resulting in different catalyst exhaust effects.

The exact nature of the deactivation is highly controversial but generally is agreed to result from the low temperature deposition of amorphous zinc and/or phosphorus compounds on the washcoat surface. These deposits cannot be removed from the catalyst, except under conditions which are likely to cause thermal deactivation (that is, very high operating temperatures).

Pore blockage has been identified as one deactivation mechanism (12). The formation on the washcoat surface of a glassy zinc phosphate layer which is impervious to exhaust gas has also been reported (11, 12). Noble metal/phosphorus interaction has not been identified as a definite deactivation mechanism. Ageing at rich air:fuel ratios in the presence of combusted ZDDP causes greater catalyst
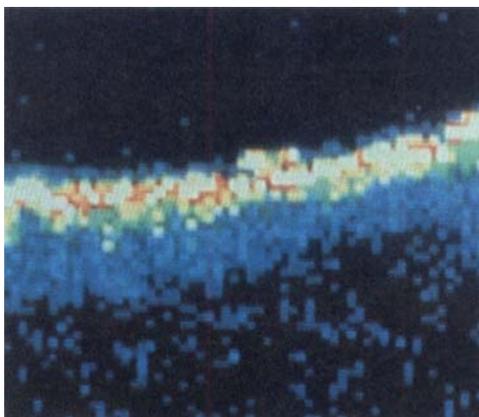
Fig. 2 X-ray microanalysis of a section through a catalyst surface shows the build-up of a coherent phosphorus layer which penetrates into the washcoat

(13, 14). More recent work examined the effect of phosphorus levels in lubricating oil on catalyst performance (14). This programme examined three levels of phosphorus, below 0.07, 0.07–0.09 and above 0.09 per cent, and studied the effect on catalyst activity by running the samples for an extended period of time on a small single cylinder engine test rig.

The study concluded that catalyst poisoning and hence loss of activity increased with increasing phosphorus content in the oil. However, oil consumption was not found to be statistically significant in affecting catalyst performance. High phosphorus oils produced dense coherent deposits on the catalyst surface, Figure 2, and penetrated the washcoat to a greater depth than low phosphorus oils which resulted in barely discernible deposits, Figures 3(a) and (b). These thick deposits act as a diffusion barrier to exhaust gases, and hence a reduction in catalyst activity occurs.

Similar deposits of phosphorus on oxygen sensors retard their response time, which could affect the closed loop control accuracy of the system after extended durability.

The study concluded that oils designed with suitable antiwear technology and reduced phosphorus content are beneficial.

deactivation than similar ageing under lean conditions (11).

Deactivation by ZDDP can be reduced by increasing the ratio of alkaline earth metal to phosphorus in engine oil additives, this is shown by the data given in Figure 1. This has the effect of decreasing the amount of phosphorus deposited on the catalyst. Such oils have been developed, and reduce catalyst deactivation without compromising engine wear

Fig. 3 Diffusion profiles for the oxides present in catalyst washcoatings contaminated by lubricating oil additives; (a) low and (b) high phosphorus content test oils. In each case phosphorus was the major element detected and it penetrated up to 30 micrometres into the washcoat, with the high phosphorus oil

It should be pointed out however that some of this work simulated very high levels of oil usage—up to five times normal—in a relatively short time. Correlation between this type of test and actual road durability is therefore difficult to establish. Other work in mixed road durability trials over 50,000 miles where oil usage was not excessive but within normal limits has suggested that there was no correlation between tailpipe emissions in the FTP75 test and phosphorus levels up to 0.1 wt. per cent in the oil. Despite these findings, it is not unrealistic to expect that lower phosphorus levels in the oil, providing engine wear is not adversely affected, must be of benefit for extended catalyst durability.

Conclusions

The main conclusions that can be drawn from this survey of the published literature, are as follows: a lead content of 3 mg per litre of fuel, should be acceptable for most driving conditions. For continual use, a fuel lead content of 10 mg/l is too high to allow vehicles equipped with three-way catalysts to meet U.S. legislated limits, or their equivalent, after 50,000 miles of use. However, levels as high as this will not regularly occur with unleaded fuel usage since in order not to exceed the maximum level of 13 mg/l at the pump nozzle the average level should be lower, and in practice is likely to be less than 3 mg/l.

Vehicle misfuelling is highly detrimental to catalyst activity even when incidents are infrequent and intermittent. The use of special filler nozzles and tank orifices make it impossible for misfuelling to happen accidentally.

Manganese derived from MMT added to fuel has shown some adverse effect on catalyst performance in mileage accumulation testing. The effect appears to be a function of temperature. No experience of MMT under European driving is available, but MMT is not expected to be used in European fuel due to the possible adverse effects on catalysts, and its toxicity in the environment.

Deactivation by engine oil derived phosphorus can be significant, especially when catalysts operate continuously under low temperature conditions, with excessive oil usage. However, well formulated lubricants and well designed catalyst systems will ensure that catalyst equipped vehicles meet the emissions standards required.

The use of catalysts incorporating platinum group metals is the most suitable method of ensuring that more severe emission levels and durability targets are met in the future.

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Protective Coatings for Turbine Components

The effects of platinum metal additions on the properties of coatings used to protect gas turbine components have been considered here previously, most recently in a brief alerting item published last July (1). Further additions to the literature have since appeared.

One of the keynote papers presented at the European Colloquium on the role of active elements in the oxidation behaviour of high temperature metals and alloys, organised by the Commission of the European Communities and held at the Institute of Advanced Materials, Petten, considered the effects of platinum on the oxidation and hot corrosion of coatings. A group of scientists from the University of Pittsburgh presented the results of their investigation of diffusion aluminide coatings on nickel base superalloys (2). Such coatings can be degraded by several different processes, and their aims were to identify the types of degradation that could be retarded by the addition of platinum to the coating, and then to establish the mechanisms by which this advantage was obtained.

They concluded that the addition of platinum to diffusion aluminide coatings improved the resistance of these coatings to the main types of corrosion encountered in high temperature service, namely cyclic oxidation, high temperature hot corrosion and low temperature hot corrosion. In the case of the last of these the addition of platinum was not as effective as for the other two, but it still gave an improvement. For effective protection, the surface of the aluminide coating had to be predominantly platinum and aluminium, and the platinum serves to promote the selective oxidation of the aluminium.

The results of a study undertaken at Solar Turbines Incorporated to evaluate the relative corrosion resistance of a range of commercially available diffusion and overlay coatings, typical of those used to protect the base metal superalloys from which gas turbine components are made, have been reported (3). The evaluation of two nickel base (MAR–M421 and IN–738LC) and one cobalt base (FS–414) superalloys was carried out after furnace testing with synthetic sea salt deposits at 704 and 899°C, temperatures regarded as crucial for low temperature and high temperature hot corrosion, respectively.

The work indicated that a silicon aluminide and platinum–chromium aluminide afforded the best protection. Recommendations for coating selection to provide optimised corrosion resistance under various conditions are tabulated.

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Meter for Food Samples

With the recent application of ohmic heating to food processing, during which an alternating current is passed through the food and sufficient heat is generated in situ to sterilise it, there has arisen a need to establish the electrical conductivity of food samples. For economic reasons, processing is usually carried out at 50 Hz, the frequency of the electricity power supply. However it is known that for foods with a high moisture content the electrical conductivity may be dependent on the frequency.

To support a study of the ohmic heating of foods being undertaken at the University of Cambridge, test equipment has been designed which enables the rapid measurement of the conductivity of food samples (F. R. G. Mitchell and A. A. P. de Alwis, J. Phys. E: Sci. Instrum., 1989, 22, (8), 554–556). The conductance meter cell and the associated electronic unit are described. Between experiments effective cleaning of the cell is essential, and it incorporates two 10 mm diameter platinised electrodes, between which the food is clamped.