The Oxidation Stability of Gear Oils in Modern Differentials; A More Hostile Environment

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SUMMARY

For many years, rear axle fluid has been capable of handling steadily increasing levels of energy transfer. However, rear axle lubricant temperatures have increased to levels that may significantly shorten the life of the fluid and threaten the durability of the differential. The effect of increased temperature is to increase the rate of oxidation of the differential fluid to the point where it no longer can serve its role of lubrication. The desirable goal of developing more dependable differential fluids suggests the development of , bench tests for screening and comparing candidate fluids.

The focus of this work is to examine the use of the Thin Film Oxidation Uptake Test (TFOUT) apparatus ASTM D4742[1]. Using a specific catalyst more appropriate for the differential fluid environment, this portion of the study was focused on to determination of the relationship between axle fluid temperature and axle fluid degradation. This paper details the development of a basic test method and its application to the oxidative stability of some commercially available gear oil formulations.

1. INTRODUCTION

Over the last few decades, the use of rear wheel drive light trucks and SUV's in North America is more common than ever before. Sport utility vehicles and light trucks now comprise over 50% of the North American automotive market. With every model change, these vehicles become more powerful and capable of carrying and towing heavier loads. It is, thus, highly important that such powerful vehicles, capable of doing more demanding work, have differential fluids matching such abilities of the vehicle.

Increasingly higher differential gear oil temperatures result as a consequence of higher horsepower engines sending greater power through smaller differentials (relatively speaking) on more aerodynamic vehicles. This trend is not new, and a number of papers have attempted to address these issues, as far back as 1983, when Schiemann et al. presented a paper on the impact of vehicle changes and the effect these changes have had on gear lubricant requirements [2]. At that time the main trend was vehicle horsepower and the airflow and cooling issues had not become as important as they are today. To fully understand the current severity trend in gear oils one need look no further than recently published papers such as Akucewich et al. [3].

In a proprietary dynamometer test closely correlated to field conditions many axles regularly exceed 150°C. Operating temperature is increasingly important to the design and engineering professionals who design axles for customer satisfaction. A premium is placed on operating temperature because of its link to axle durability. Thus, the thermal and oxidative stability become considerably more important in this environment.

From an automotive OEM perspective, the desired solution to increasing axle temperatures lies in controlling temperature by improved axle design, proper vehicle packaging and maintaining adequate airflow for cooling. As engineering and design groups attempt to lower the axle operating temperature, it is necessary to clearly understand the effect that temperature has on gear oil oxidation stability. Lower operating temperatures will ultimately lead to a higher performing fluid with longer drain intervals. This will benefit the customer and the environment. Additionally, the formulation of gear oils requires a balance between durability concerns and fuel economy. Operating temperature is a key factor in this relationship.

There have been many lubricant oxidation tests developed in the past and all of these test methods posses certain advantages and disadvantages relative to each other. The goal of this research was the design of a simple, relatively precise but comprehensive bench test specific to automotive axle oil that would clearly display a fluid's oxidation characteristics under variable conditions simulating operation of the differential.

The test would have to accomplish the following objectives:

- Differentiate gear oils according to their relative oxidative stability
- Correlate to real world vehicle use
- Be capable of indicating the oxidation characteristics of both base oils and additive components

To accomplish these objectives, the test should measure:

- Oxidation
- Viscosity change
- Deposit formation

2. BACKGROUND

While there is a large body of literature on the oxidative stability of lubricants in general, not surprisingly, the body of literature on the oxidative stability of automotive axle oils is relatively small. Much of the available literature tends to focus on either engine oil or transmission fluid. Much of the transmission oxidation work dates back to the original development of the ABOT at Ford Motor Company in the late 1970's [4,5]. The oxidative stability of engine oils has been characterized in a diverse and extensive manner the last 50 years.

Choice of the Thin Film Oxygen Uptake Test

On the basis of the need for versatility and precision, the development and application of the Thin Film Oxygen Uptake Test (TFOUT), (originally conceived by Hsu and associates for determining the acceptability of re-refined base oils for use as engine oils during the oil embargo of the 1970's [6]) was chosen. This was followed by other work directed at improving the instrument and applying it to different areas of need [7,8] One paper in particular provided good insight into the catalyst package and the various mechanisms of oxidative degradation found in the TFOUT [9] Discussion of the interaction effects between the nitro compounds and some amine antioxidants used as supplemental inhibitors used in engine oil formulations and the proposed mechanisms provided valuable insight regarding modification of this test for the work presented in this paper.

3. TEST APPARATUS AND PROTOCOL

Test Apparatus – The Designed TFOUT

The original work on the TFOUT used the Rotating Bomb Oxidation Test (ASTM D 2272[10]) as a prototype test base. Although prototype instruments continue to be widely used for the TFOUT test, an apparatus designed for determining TFOUT values was created. Information on this instrument and studies conducted with it are available in the literature [11, 12].

As previously noted, the TFOUT test was originally developed to monitor batch-to-batch variation of re-refined base stock formulated engine oils under the recycled oil program of RCRA and to simulate the oxidation response in the Engine Oil Sequence IIID test (and later the IIIF test). As in most benchtop test development efforts the purpose of this work was a reduction in the capital and time commitment of vehicle or dynamometer testing. The same purpose was intended in the present development and application of the apparatus.

Protocol

Protocol for Engine Oils - The TFOUT protocol for engine oils requires that 1.5 g of the test oil be mixed with a complex catalyst in a special short beaker and the beaker placed in a pressure vessel. The pressure vessel is then pressurized with pure oxygen and placed in a heated environment controlled at 160°C. At this point the beaker is rotated at 100 RPM as the test starts and the internal pressure of the pressure vessel continuously monitored. The test continues until the oil's resistance to oxidation suddenly fails and the oil rapidly oxygenates the oil producing a rapid drop in the internal pressure of the vessel. This is called the "break point" and the time to reach this point is called the induction time or "break time".

Modified Protocol for Gear Oils - The study presented in this paper is based on the TFOUT protocol for engine oils but modified for gear oils by using ferric naphthenate as the sole component of the catalyst. Catalyst composition and concentration were based on vehicle contamination found in typical DaimlerChrysler vehicles under various service conditions and mileage.

In the TFOUT test for engine oils, the test is always run until the break point occurs. However, in running the TFOUT protocol for gear oils, the protocol also included stopping the test at any point before or after the break point. Temperature ranges were based on data obtained during DaimlerChrysler vehicle testing. Time range was base on both severity of results and reasonable test duration.

Tests for Oxidation Level - The oxidative stability of gear oils can be monitored or defined in several ways:

- viscosity change
- acid number change
- oxygen consumption
- formation of oxidation products (sludge, varnish, suspended or solubilized oxidation product etc…

The interrelationship between oxidation and its various manifestations and the lubricant's ability to function is reasonably assumed to be significant.

Of these oxidation level tests, FTIR (Fourier Transform InfraRed) and Acid Number were chosen for first studies as being directly related to . The later is a well-known standard ASTM Test Method D664[13] and requires little comment here except to note that it is a titrimetric method using electrode potentials to determine the titration end point.

The degree of oxidation was determined using the Fourier Transform Infrared (FTIR) subtraction of the FTIR spectrum of the oxidized oils from the fresh oil to determine change in oxidation in units of absorbance per unit length of light transmission, A/cm. This allowed the determination of oxidation level with only a thin film of oil on the ATR plate, which is important considering that the amount of oxidized oil from the TFOUT test is limited.

4. MATERIALS

Table 1 lists some of the properties of the gear oils used in these studies. Included are factory-fill oils and commercial products.

Table 1: Differential Oils Tested

Axle Oil	Base Oil	40°/ 100°C Kin Vis.	B % Wt.	P % Wt.	Ni% Wt.	S % Wt.
Oil C 75W-140	PAO*	185/ 25.0	0.024	0.141	0.09	2.39
Oil B 75W-90	Group III	102/ 14.0	0.026	0.133	0.105	2.24
Oil B+FM** 75W-90	Group III	94/ 13.8	0.026	0.185	NA	2.09
Oil A 75W-90	PAO	144/ 19.1	175	3673	3182	2.34

*Poly-a-olefin; ** FM: friction modifier (added at 5%)

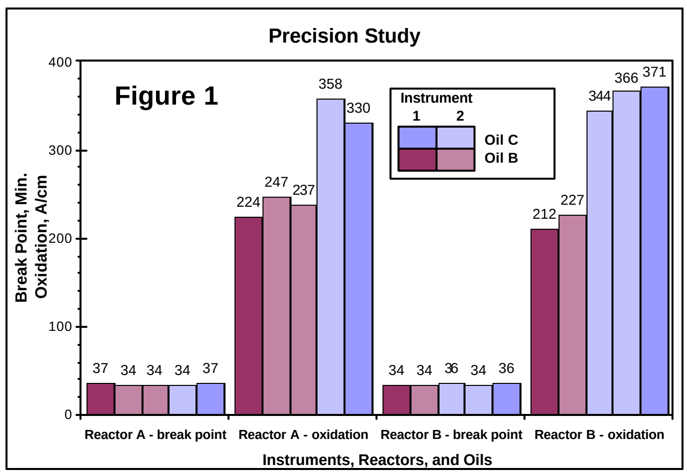
It should be noted that only Group III and Group IV base oils were used in these gear oils since it was assumed that traditional solvent-neutral base oils would fail to provide the oxidative stability required to meet the requirements of the differential.

5. PRECISION STUDY

The precision of a bench test is an important criterion, particularly in oxidation studies. In order to test the precision of the modified TFOUT protocol and apparatus, two factory-fill gear oils were run five times using two modified TFOUT instruments. Each oil contained a catalyst level of 1.5% ferric naphthenate at 170°C. The test was continued for 60 minutes at which point the test was stopped, the oxidized oil recovered and the degree of oxidation determined. Results are shown in Table 2 and Figure 1.

Table 2: Precision of the Modified TFOUT Test

Oil	Unit/Cell	Break Point	Oxidation Level Absorption/cm
Oil B	1A	37.0	224
Oil B	1B	34.0	212
Oil B	2A	34.0	247
Oil B	2A	34.0	237
Oil B	2B	34.0	227
Oil B Avg.		34.6	229.4
Oil B Std. Dev.		1.3	11.9
Oil B % Dev.		3.7	5.2
Oil C	1A	37.0	330
Oil C	1B	36.0	371
Oil C	2A	34.0	358
Oil C	2B	34.0	366
Oil C	2B	36.0	344
Oil C Avg.		35.4	353.8
Oil C Std. Dev		1.2	15.0
Oil C % Dev.		3.4	4.2



The data show that repeatability within and between instruments is good; particularly considering the difficulty normally associated with reproducing oxidation levels in most instruments.

It was important to note that the standard deviation was, on average, 3.6% for the break point and that the difference in average break point of these two oils was 2.3%. In other words, despite the good precision, it would be difficult in this case to differentiate these oils according to their break points. Oxidation, in absorption units per centimeter, on the other hand had an average standard deviation of 4.7% while there was a difference in oxidation number at break point of 42.7% between the two oils. This indicates clear and statistically significant differentiation by oxidation level between these two oils.

However, it should be mentioned that the FTIR values shown in Table 2 were taken from TFOUT tests that were run for one hour although the break points came after only about 35 minutes. This indicates that oxidation continuing after the break point may be different for different differential fluids.

6. STUDIES OF MULTIPLE FACTORS ON TFOUT RESULTS - TAGUCHI TEST RESULTS

Taguchi Matrix Analysis Protocol

The primary need in this study was to establish and understand the relative contribution of the factors responsible for gear oil oxidation. The most important factors in the differential environment were considered to be temperature, time of exposure, and catalytic influence.

One way of evaluating the relative importance of variables is to use matrix analysis of which the techniques of Genichi Taguchi [14] have been highly developed over the last 50 years. Taguchi's technique permits the determination of the contribution of each of three variables to a test method with only four tests required. To quickly establish a reasonable point from which to initiate testing, a Taguchi study using these three critical variables of time, temperature, and catalyst level was set up using the modified TFOUT to generate oxidation of the sample.

Summing simply, the method uses orthogonal arrays similar to partial factorial experimental designs to permit determination of the relative contributions of the number of factors chosen to produce a measured result such as a TFOUT break point[15].

In the Taguchi L-4 matrix analysis, the influence of all three parameters must add up to 100%. This is very helpful in evaluating the relative magnitude of influence of each parameter. In comparison, the TFOUT is most meaningful for absolute differences among oils.

In setting the parameters for a Taguchi analysis, a sufficiently high and low value is chosen for each parameter as shown in Table 3 for the three parameters of catalyst concentration, exposure time, and test temperature. Past cursory gear oil analysis indicated that exposure times may be relatively low compared to engine oils and this tended to dictate this parameter in Table 3.

Table 3 – Values Set in Taguchi Test Protocol #1

Parameter	Low Value	High Value
Fe Concentration	0.5%	1.5%
Time of Exposure	30 Min	90 Min
Temperature of Test	120°C	170°C

Additionally, automotive industry trends helped form the test parameters. For example it has been observed that some oils under high temperature conditions tend to lose their capability at different rates, for this reason the upper temperature limit was set above the commonly recognized problem temperature of 150°C.

Taguchi Study #1: Comparison of Oil C and Oil B

The two test oils initially chosen were those used in the precision study, Oils B and C. It was thought that these results would provide reference information for the studies to follow since, as previously mentioned, both were factory-fill oils. Oil B as seen in Table 2 is a Group III SAE 75W-90 while Oil C is a full synthetic SAE 75W-140. Both of these oils have been used extensively in industry and different thermal and oxidative stability trends have become apparent in the field.

To compare the effects of the three factors of catalyst concentration, exposure time, and exposure temperature, two sets of test sequences were needed using the parameters of Table 2. Table 4 shows the information.

Table 4 – Taguchi Test # 1, Part 1, Oil B Compared with Oil C

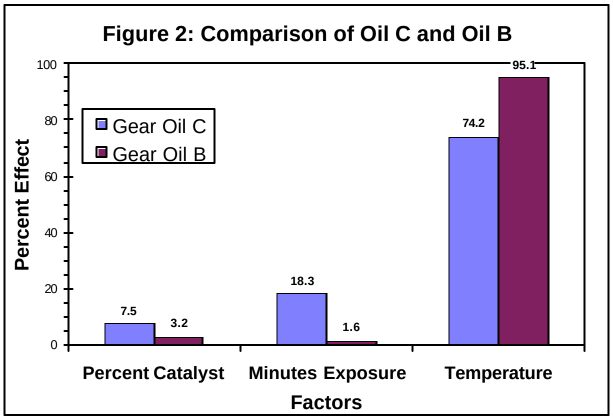
Test Sequence	Catalyst Level, %	Exposure Time, min	Exposure Temp., °C	Oxidation, A/cm	
				Oil C	Oil B
1	0.5	30	120	7	10
2	0.5	90	170	108	53
3	1.5	30	170	53	55
4	1.5	90	120	19	22

As would be expected, the higher levels of temperature and exposure time have a greater effect on oxidation than do their low-level values. It is evident that the two gear oils respond differently to the conditions applied. The difference is particularly notable in the Test Sequence 2 conditions of high stress from exposure time and temperature.

It is interesting to note that under the less severe conditions of Test Sequence 1, Oil C seems to slightly outperform Oil B, but at higher temperatures and longer exposure times of Test Sequence 2, Oil B significantly outperforms Oil C. More significantly, it appears that the base oils and additive packages of the Oils B and C in this initial study will protect the oils from significant oxidative degradation at lower temperatures, but as exposure time and temperature increase, the oxidative stability of the oils is significantly diminished. Oxidation has been increased by a factor of more than 10 for Oil C and by a factor of 5 for Oil B in going from the less severe to the more severe exposure condition in the modified TFOUT.

Taguchi Matrix Analysis - Using the power of matrix analysis, the percent influence of each of the three parameters can be determined. As mentioned earlier, the influence of these parameters must add up to 100%. In other words, while other parameters not included in the Taguchi L-4 study may be influential, the matrix analysis gives the percent contribution of the three factors composing the study. Larger matrices can be used in Taguchi analyses but the L-4 matrix was considered adequate in this work.

The two Taguchi L-4 experiments composed the first study whose results are shown in Figure 2. Summation of the percent contribution values adds up to 100% and this provides a relative measure of the importance of each variable on every gear oil.



The Taguchi L-4 test clearly shows that exposure temperature has the greatest influence on the oxidation levels of both oils under this Taguchi protocol.

Moreover, to the degree that the data are sensitive to small effects of the parameters, Oil B was less sensitive to catalyst concentration and test temperature than Oil C. With considerably less response to catalyst concentration and time of exposure, temperature was, by far, the greatest influence regarding oxidation. However, it must be kept in mind from Table 4 that the actual oxidation level of Oil B was about half that of Oil C for the more severe test conditions.

The information does not distinguish whether the relatively different oxidation stability is a result of base oil or additive composition. That is, there may be a comparatively similar oxidative stability of the PAO base oil of Oil C and the Group III base oil of Oil B, but a difference in additive content or capability of resisting oxidation. Alternatively, perhaps there is a difference in base oil susceptibility to oxidation that the additives cannot ameliorate at higher temperatures. For example perhaps the PAO base oil may start to degrade at a much higher rate above some temperature level below 170°C even though the break points are the same at 150°C as shown in Table 2.

Taguchi Study #1, Part 2: Comparison of Oil B Plus Friction Modifier to an Experimental Oil A

Friction modifiers are present in gear oils to obtain smooth engagement of the clutch plates in the limited slip differential mechanism. The friction modifiers used are also susceptible to oxidation and lose this ability. Resultant "stick-slip" clutch engagement produces highly undesirable shuddering in the motion of the vehicle.

On the basis of the information gained from the previous Taguchi study of Oils B and C, it was of interest to appraise Oil B plus a friction modifier against Oil A, an experimental oil of similar SAE 75W-90 classification with PAO base oil. (Gear Oil A contains friction modifier as part of its formulation.) The variables and test parameters were kept the same as in the previous comparison of Oil C and Oil B (see Table 3). In this case however, both test oils are SAE 75W-90 gear oils and, as noted, both gear oils contain friction modifiers. Table 5 shows oxidation results.

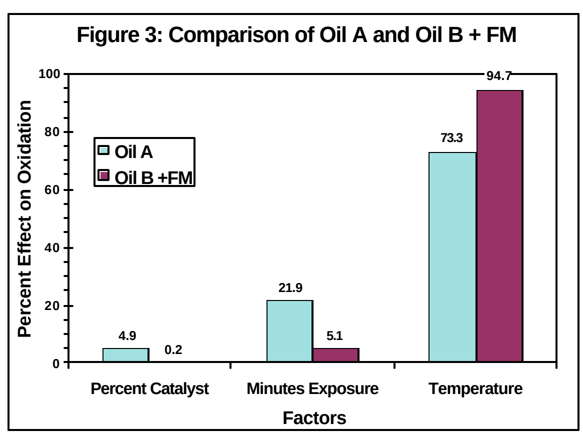
Table 5: Taguchi Test # 1, Part 2, Oil B +FM Compared to Oil A

Test Sequence	Catalyst Level, %	Exposure Time, min	Exposure Temp., °C	Oxidation, A/cm	
				Oil B +FM	Oil A
1	0.5	30	120	11	12
2	0.5	90	170	64	87
3	1.5	30	170	56	48
4	1.5	90	120	23	26

Once again, under conditions of high temperature and extended exposure time, Oil B outperforms the comparison oil, Oil A. However, within the limitations of the precision for the TFOUT gear oil protocol, comparing results in Table 5 to Table 4 for Test Sequence 2, it appears that the addition of 5% friction modifier has made Oil B somewhat more susceptible to oxidation (with friction modifier, 64 A/cm vs. without friction modifier, 53 A/cm).

Taguchi Matrix Analysis - Figure 3 compares Taguchi matrix analysis of Oil B+FM with Experimental Oil A. It is apparent that Oil A shows greater dependence on exposure time and catalyst which results in a lower value of temperature dependence. This is similar to Oil C tested in Taguchi #1 Part 1, also a PAO based gear oil. On the other hand, Oil B has again shown itself more dependant on temperature and less dependent on catalyst concentration and exposure temperature than both of the other oils tested at that point of the studies.

Taguchi Study #1 Part 3: A Comparison of Oil B and Oil B With Friction Modifier



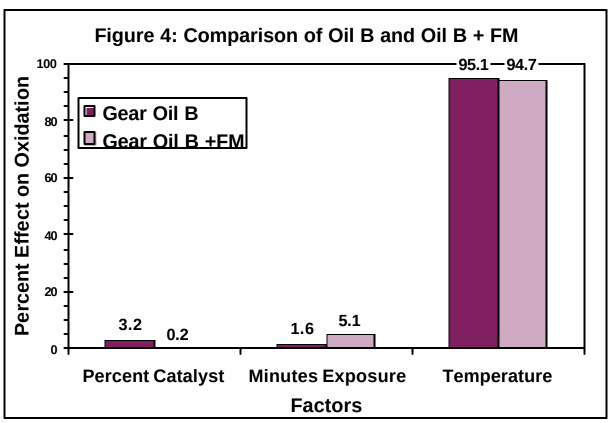
With the clear value of obtaining Taguchi information comparing different gear oils, it was considered of interest and importance to compare Oil B with and without the presence of friction modifier. The primary purpose of this comparison was to gain a better understanding as to the role FM plays in the overall oxidation stability of a gear oil formulation. Table 6 shows the results of the study.

Table 6: Taguchi Study # 1, Part 3, Oil B Compared to Oil B Plus 5% Friction Modifier

Test Sequence	Catalyst Level, %	Exposure Time min	Exposure Temp °C	Oxidation, A/cm	
				Oil B	Oil B +FM
1	0.5	30	120	10	11
2	0.5	90	170	53	64
3	1.5	30	170	55	56
4	1.5	90	120	22	23

It appears from Table 6 that the friction modifier has only a small or moderately negative effect on oxidative stability at low temperatures. This effect appears to become more pronounced as temperature and its duration increase as shown by Test Sequence 2. Otherwise, with less exposure time at higher temperature (Test Sequence 3) or longer exposure time at lower temperature (Test Sequence 4), there is little or no effect.

Taguchi Matrix Analysis- Figure 4 shows the Taguchi matrix analysis results and the similarities between the two gear oils are evident. Slightly greater sensitivity to exposure time by Oil B plus friction modifier is shown and reflects the influence of Test Sequence 2.

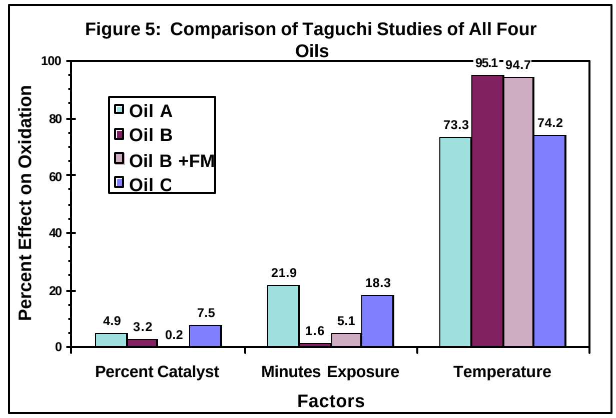


Summary of Taguchi Study #1: A Comparison Of All Four Gear Oils

Taguchi Matrix Analysis - Table 7 and Figure 5 summarize the TFOUT-Taguchi information obtained thus far. One point of view is that the information can viewed as a ranking of the relative performance of the four gear oils. From this view, it appears that Oil B with or without friction modifier is most resistant to oxidation at high temperatures followed in order by Oils A and C.

Table 7 – Taguchi #1 Summary: Comparison of the Four Gear Oils Tested

Test Sequence	Cat. %	Exp. Time	Exp Temp.	Relative Oxidation Level, A/cm			
				Oil C	Oil B	Oil B +FM	Oil A
1	0.50	30	120	7	10	11	12
2	0.50	90	170	108	53	64	87
3	1.50	30	170	53	55	56	48
4	1.50	90	120	19	22	23	26



It is also interesting to note that in Sequence 2 the two PAO-based products, Oils A and C, show markedly greater levels of oxidation. Still, the role of the additives and their respective levels of oxidation resistance remains a highly relevant question.

7 TAGUCHI STUDY #2: EFFECTS OF APPLYING A 150° TO 170°C TEMPERATURE RANGE

To gain additional information, the previous information at 170°C was used in conjunction with two tests at 150°C. By merely substituting two tests at 150°C for each gear oil and replacing the four sequences at 120°C, a new Taguchi comparison could be made. This set of Taguchi tests was identified as Taguchi Study #2. Essentially, the temperature variable lower limit was raised from 120°C to 150°C.

The reasoning behind this portion of the study is twofold. First, reducing the temperature difference between the highest and lowest temperature parameter was expected to sharpen the parameters of exposure time and catalyst concentration relative to the temperature parameter. This would be expected to alter the relative ranking of all three factors and give greater insight into the roles of the catalyst and exposure time. Secondly, given the relative dominance of temperature, raising the minimum test temperature would likely give greater differentiation amongst the oils tested.

From Tables 4, 5, and 7, the test sequences at 120°C were replaced by information gained at 150°C and the data reanalyzed using the Taguchi matrix technique. Comparison of the four oils is shown in Table 8.

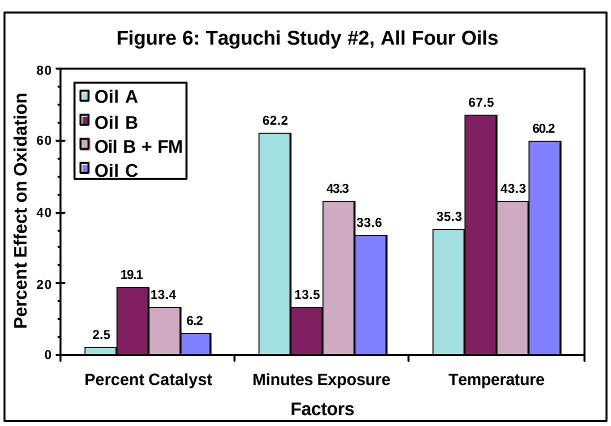
Table 8 – Taguchi Study #2: Data on Four Gear Oils Applying Temperatures of 150° and 170°C

Test Sequence	Catalyst Level %	Exp Time Min	Exp Temp ° C	Relative Oxidation Level, A/cm			
				Oil C	Oil B	Oil B +FM	Oil A
1	0.50	30	150	18	19	28	30
2	0.50	90	170	108	53	64	87
3	1.50	30	170	53	55	56	48
4	1.50	90	150	40	42	56	56

The overall ranking of the fluids according to oxidation in each of the individual sequences appears to be similar to that in the first Taguchi study. It should be noted that only Sequences 1 and 4 are affected since these are the only tests conducted at 120°C in the first Taguchi matrices shown above in Table 8. Comparing these results to those in Table 7, all four oils show an increase in oxidation in the test sequences conducted at 150°C, as would be expected. The two oils that contain FM however, are disproportionately affected by the increased temperature parameter.

The effect of the adding 5% of a friction modifier is interesting and the new study at 150°C adds information. Apparently, the effect of the added friction modifier makes the fluid 35% to 45% more vulnerable to oxidation at 150°C (depending on the catalyst concentration and the exposure time). However, at 170°C, little further effect is found indicating that the most of the oxidation response of the friction modifier occurs at or before 150°C (again depending on catalyst concentration and exposure time). Essentially it appears that the FM undergoes significant oxidation between 130°C and 150°C.

Figure 6 shows the results of the Taguchi matrix analysis



of these data.

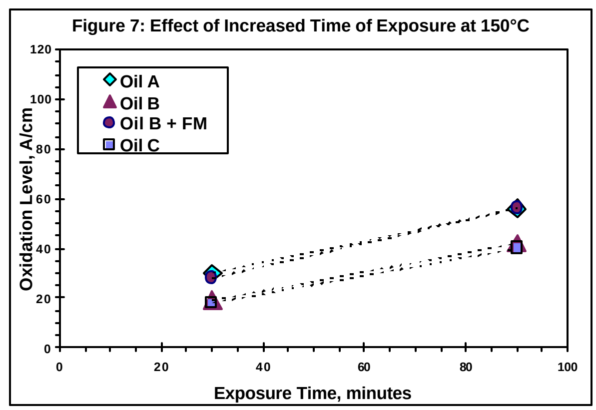
Comparing this figure with Figure 5, the power of Taguchi analysis (as practiced in this paper) comes into perspective. By decreasing the temperature interval between the lower and upper values from 120°-170°C to 150°-170°C, influence of the exposure time and even the catalyst content now becomes evident.

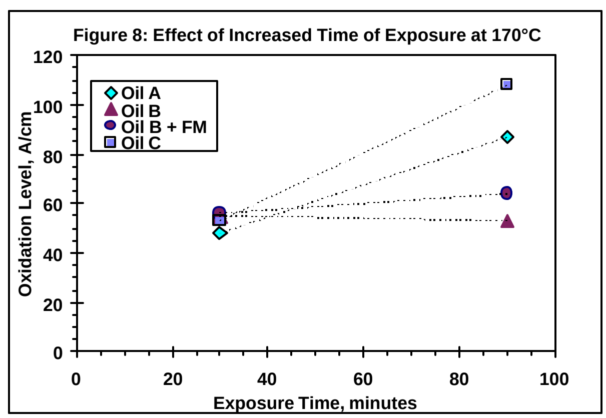
Oil A is shown to be insensitive to the catalyst content but highly sensitive to exposure time and exposure temperature. Oil B, in contrast, now shows somewhat more sensitivity to the catalyst concentration but is relatively insensitive to exposure time while very sensitive to exposure temperature. Again, in contrast to Oil B, Oil B with 5% friction modifier is somewhat less sensitive to the catalyst, much more sensitive to exposure time and equally sensitive to exposure temperature.

Oil C shows essentially no increase in catalyst response, but an increase in sensitivity to exposure time (at some sacrifice of its prior evidence of sensitivity to temperature). The latter effect must still be viewed as high for Oil C and simply a value accommodating the increase in sensitivity to exposure time (since, as previously noted, the total of the three effects must be 100%).

Analysis of Oxidation Response at 150° and 170°C

The information obtained in the Taguchi studies can be applied on an absolute basis if desired. For example, in Figures 7 and 8, the experimental results obtained at 150° and 170°C can be examined for further information.





In Figure 7 it is shown that at 150°C, the four Oils A, B, B+FM, and C all have similar rates of oxidation. The two oils containing friction modifiers, Oils A and B+FM, are similar in initial oxidation and in oxidation rate. At somewhat lower initial oxidation levels, oxidation rate is also the same for non-friction-modified Oil B and Oil C.

However, in Figure 8, with data taken at 170°C, all four of the gear oils have reached a level of equivalent oxidation at 30 minutes. However, neither Oil B nor Oil B+FM changes substantially with a further 60-minute exposure. In comparison, Oil C and Oil A continue to show oxidation susceptibility at 170°C. When displayed in this manner, the degree to which all of the gear oils respond differently to temperature is striking.

Thus, it would seem that for Oils A and C, the additives or the entire formulation (including base oils) continue to absorb oxygen beyond 30 minutes. It may be that the deposit-forming tendencies of the gear oils are held in abeyance by this additional oxygen-absorbing ability. This consideration led to the desirability of determining the deposit forming tendencies of the gear oils -- perhaps by using the weight of deposits that form in the glass beakers after a measured time of test coupled with non-deposit cleaning techniques to preserve the deposits and permit weighing.

Taguchi Study #3: Effect of Constant Catalyst Level

The previous study of the actual oxidation levels of the four gear oils provided insights into the relationships of the parameters of exposure time and temperature but with the catalyst as a variable parameter. This made it necessary to make conditional statements about exposure time and temperature parameters. The next portion of the study eliminated the catalyst as a variable.

Table 9 presents the data obtained by running those TFOUT tests which would bring the concentration of catalyst in all four oils, Oil A, B, B+FM, and C, to a value of 0.5%. These are the results obtained using the data obtained at a minimum temperature of 150°C on the four gear oils.

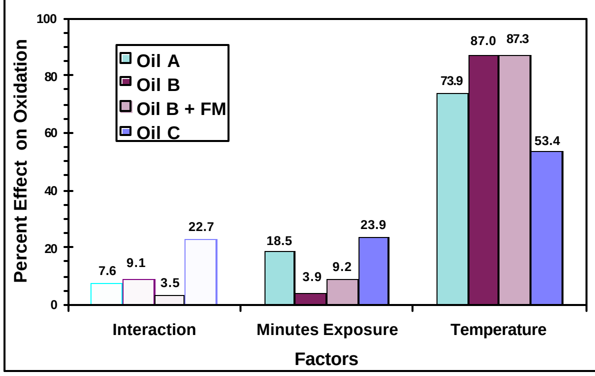
Table 9 – Taguchi #3 Results Using Constant Catalyst Level

Test Sequence	Cat %	Exp Time Min	Exp Temp ° C	Relative Oxidation Level, A/cm			
				Oil C	Oil B	Oil B +FM	Oil A
1	0.5	30	120	7	10	11	12
2	0.5	90	170	108	53	64	87
3	0.5	30	170	28	34	43	46
4	0.5	90	120	8	6	16	21

Comparing Table 9 to Table 7, Sequences 3 and 4 are the two that are affected (since Sequences 1 and 2 were already obtained at a catalyst treatment level of 0.5 %). Essentially, comparing Sequences 3 at 170°C and 4 at 120°C in both tables shows that tripling the catalyst level to 1.5% doubles the response of Oil C and Oil B but has little effect on Oil B+FM or, particularly, Oil A. A peculiar response when one considers oils B +FM and Oil A both contain friction modifier.

Choosing a constant catalyst level as a specific parameter produces an interaction parameter showing how the

Figure 9: Constant Catalyt Level Effect on Four Oils



parameters of exposure time and temperature affect one another. These values are also shown in Figure 9.

Comparing this data of Figure 9 to that of Figure 5 suggests that, while the influence of exposure time has been somewhat increased for all four oils and the interaction effect for Oil C is particularly strong, nonetheless, the influence of temperature is still the dominant parameter. The similarity between the relative rankings of oils in the two figures is notable as well. This would seem to indicate that, between 0.5% and 1.5%, the catalyst level is not particularly critical and that 0.5% is an adequate level for further work.

7. CONCLUSIONS AND DISCUSSION

As mentioned previously, this study was initiated to gain a greater understanding of the roles of time and temperature on gear oils in the presence of the iron that is integral to the operating environment of the differential. Of the various oxidation tests available, a modified TFOUT apparatus was chosen around which to investigate these relationships and, if possible, to ultimately serve as the bench test for qualifying gear oils.

A initial study of precision of the proposed protocol showed good precision using two instruments and operators.

Field and dynamometer experience with Oils B and C were reflected in the differences shown in the TFOUT bench test equipment and protocol. However, the understanding of the data was further illuminated by applying the Taguchi matrix analysis technique in which chosen variables could be quickly sorted out as to their influence on gear oil resistance to oxidation. The results clearly indicate the degree to which temperature, time of exposure, and iron catalyst concentration has on each of the four oils made part of the study.

In addition the analysis elegantly illuminated a relatively simple but important point: the lubricant engineer needs to carefully consider not only expected in vehicle axle operating temperatures but peak temperatures as well, when specifying the most appropriate lubricant technology for use in that vehicle.

Operating temperature had the largest effect on gear oil oxidation, followed by the time over which that temperature was applied. Between the limits of 0.5% and 1.5%, the catalyst concentration effect was relatively minor and 0.5% concentration of catalyst seemed acceptable. Not surprisingly, there were considerable variations among the four gear oils studied in their response to the factors of time and temperature. Although only one additive response was investigated in this preliminary study – that of a friction modifier – its influence on oxidation susceptibility was apparent but not large.

Essentially, the study indicated that the modified TFOUT protocol and instrument will provide a good foundation for further study.

A logical extension of this work would be to look at the effects base oils, additives and friction modifiers have on oxidation. Given the wide range of additive technology available for gear oils, it is presently difficult to assess formulation differences and how they contribute to product performance.

Friction Modifier Response to Oxidation and Thermal Degradation

From the present data, it appears that the particular friction modifier evaluated has only a small or moderately negative effect on oxidative stability at the temperatures studied. However, although the friction modifier appears to have a relatively small effect on oxidation susceptibility, this says nothing about its function as a friction modifier.

Friction modifiers are typically complex mixtures containing a linear amine salt of an acid phosphate. It is commonly believed that the friction modifier function is chemical adsorption of the polar moiety onto the metal surface with the hydrocarbon tail hanging off of the metal surface and reducing the friction [16]

Trace amounts of unreacted amine or phosphate compounds or the friction modifier itself may contribute to the oxidative instability of the oil by different mechanisms at different temperatures. It has been considered that perhaps the friction modifier chelates the metal contaminants or other oxidation products in solution. Friction modifier degradation may affect axle durability through sludge and deposit formation resulting from relatively low thermal stability. The relationship between the function of the friction modification and oxidative stability is not yet defined and could reasonably be part of a future bench study.

Base Oil Factors

Base oil plays a significant role in the oxidative stability of the lubricant formulation. The majority of the literature suggests base oils affect oxidation in two ways 1) through the presence of impurities and 2) through the inherent level of stability of the base oil type.

The various grades of base oil possess different inherent oxidative stabilities. Base oil degradation is often measured by change in kinematic viscosity. This change is typically caused by two processes: 1) chain scission of the Viscosity Index Improver (a mechanical shearing effect), and 2) cross linking and polymerization of the base oil and other components as a consequence of oxidation. In bench testing it is assumed polymerization of the base oil will be the predominant mechanism.[17] although it is possible to introduce chain scission as a precondition. Given the current worldwide trend to use Group III and PAO base oils in automotive, SUV and light truck applications some additional gear oil oxidation work focusing in on base oil would provide valuable insight.

Additives

Additives and additive packages are of considerable importance in imparting oxidation resistance. The literature shows that at higher temperatures, additives and additive packages have varying degrees of effectiveness in inhibiting oxidation [17].

Some of the more common additives that contribute to oxidative stability include zinc di-organo di-thiophosphates (ZDDPs), sulfates, hindered phenols and amines. More relevant to gear oils, the commonly used sulfur-phosphorus chemistry contains a good degree of inherent anti-oxidation effects. Thus, often it is not necessary to add additional anti-oxidant chemistry[18]. However, different sulfur- phosphorous molecules have different degrees of stable at different temperatures.

For example, some interesting work has shown clear differences among the anti-oxidant capabilities of oils using different oxidation inhibitors, hindered phenol antioxidants compared to those using only ZDDPs. Hindered phenols tend to degrade before depletion whereas ZDDP gives protection until complete depletion[19]. Thus, as mentioned, a study of the components of gear oil formulation regarding oxidation inhibition would provide valuable insight into both the current best practice and the optimized axle formulations of the future.

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