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TBN Retention - Are We Missing the Point ? Diesel Engine Lubricant Characterization Using Multiple Used Oil Analyses

W. van Dam, D.H. Broderick, R.L. Freerks, V.R. Small, and W.W. Willis
Oronite Global Technology

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ABSTRACT

Historically, the characterization of fresh and used diesel engine lubricants has been based on a limited number of analytical techniques. One of the most important analyses has always been the Total Base Number (TBN) measurement. Although the TBN measurements are informative, easy, and quick, it can be misleading to base the judgment of an oil's performance solely on this one criterion.

This paper offers some observations from a field test, showing that some detergent approaches gave unacceptable performance even though the TBNs were at an acceptable level. It is hypothesized that some detergents do not effectively neutralize all acidic species present in the lubricant, and thereby thus reserve their own base, which shows up as TBN, while in fact the oil may no longer provide sufficient any protection against bearing corrosion. This hypothesis is supported with bench and engine test data. It is concluded with a recommendation that, at a minimum, Total Acid Number (TAN) measurements be included in any analysis. Where time and cost allows, wear metals content, oxidation, soot content, and viscosity should also be evaluated.

This hypothesis is supported with bench test data. Finally, in addition, it is demonstrated that several of the API PC-7 engine tests show the same phenomenon as our field test, suggesting that these engine and field tests do indeed correlate with the field.

INTRODUCTION

Today's market for lubricants offers a wide range of products for to lubricate internal combustion engines. Many analytical techniques exist to characterize the different lubricating oils. These are necessary to express the uniqueness of these products and/or simply to confirm their compliance with the specifications for a certain application. Among the most frequently used common characterizations are the viscosity grade, the total base number (TBN), the sulfated ash level, and the metals contents. Historically the level of acid neutralizing base, or TBN, has always been relatively high for heavy duty diesel engine oils, because of the high sulfur content of diesel fuel. Recently, Today, many governments have mandated the use of low sulfur diesel fuel for diesel engines. Normally, such a mandate would be expected to, which would lower the TBN requirements (1-4);, while at the same time on the other hand, however, there is a drive towards extended service intervals (ESI)(5), which tends to push would lead to a higher TBN requirement higher. As a result of these opposing/contradicting demands, many of the TBN requirements are currently being reconsidered.

In addition to the use of TBN for the characterization of fresh engine lubricants, TBN is also used to judge the condition/quality of used oils. In many field applications, where extended service intervals are used, TBN is measured to monitor used oil quality used to determine the status of a used engine oil, just because it is an easy and quick test to run. This widespread use of TBN grew out of the days when 1) today's sophisticated oil analysis methods were not available, and 2) much higher sulfur fuel was in use resulting in relatively stronger acids in the used oils. Several engine builders (OEMs) have set TBN limits/measurements for the condemning limits of the used oil. One OEM suggests that a reduction in TBN (D-4739) to one-third of the initial value provides a guideline for the drain interval (6). Another OEM requires fresh lubricants with a minimum TBN (D-2896) level of at least 20 times the fuel sulfur level for pre-chamber engines, or 10 times the fuel sulfur level for direct injection engines (7,8), and, furthermore, suggests a condemning limit of half of that. In general, TBN is often used to describe the quality of fresh oils and to monitor the remaining life of fresh and used engine oils.

An issue that further complicates the TBN question, is the fact that two commonly accepted measurement methods are used: 1) ASTM D-2896, often mostly used for fresh oils, and 2) ASTM D-4739, which is often used for used oils. The two methods are similar in that they involve adding a measured amount of acid to the oil until all of the base has been consumed. The TBN is calculated from the amount of acid required to completely neutralize the lubricant. The difference between the two methods comes in the choice of the acid used, and solvent in which the oil is dissolved to run the test. D-2896 uses a stronger acid than D-4739 and a more polar solvent system. The combination of a stronger acid and a more polar solvent results in a more repeatable method, and insures that all of the base present is measured. For some lubricant additive types, D-4739 does not measure all the base that is present. Table 1 shows some typical values for the ratio of TBN by D-4739 / TBN by D-2896 for a number of additive types. In general, D-4739 gives a lower result, but the difference between the two methods is not consistent. The D-4739 results are low for all ashless additives, especially for some amine oxidation inhibitors. Regardless of these differences, we have chosen to show only TBNs by D-4739 as it has become the accepted procedure for used oils.

Table 1
Difference Between ASTM D-2896 and D-4739

Additive Type	D-4739 / D-2896 Ratio (Typical Value)
Phenate Detergent	0.96
Sulfonate Detergent	0.96
Ashless Dispersant	0.48
Amine Antioxidant	0.00

Differences between the two TBN methods make the interpretation of its results difficult, and yet often, the TBN analysis is in many cases used as one of the most commonly used important means for characterizing a lubricant. In this paper a number of cautionary observations from bench, engine, and field testing will be discussed to:

1. Demonstrate the limitations of the approach of using only TBN only measurements.
2. Show the importance of several a number of related methods for the analysis of new and used oils analysis methods.
3. Underline that a thorough characterization of a used engine lubricant requires a complement set of analyses, consisting of TBN, TAN, wear metals content, oxidation, soot content, and viscosity.

FIELD TEST OBSERVATIONS

FIELD TEST DESCRIPTION - In an ongoing field test that is currently running, a number of different detergent technologies are being compared at equal dosage in the same baseline formulation. The test fleet uses Cummins N-14 engines to power the 65 tons GVW units, which run fully loaded all of the time. The test oils are rotated through the test units which. The units are run until the oil level drops 2 gallon below the maximum certain mark, and no oil is added during the drain period. This approach allows the units to accumulate 20 to 25 thousand miles without disturbing per drain period without any additions of fresh oil, which would disturb the oil aging process. During the drain period oil samples are taken and analyzed for, among others, TBN, TAN, and wear metals.

TEST RESULTS AND DISCUSSION - The results of the TBN analyses are shown in Figure 1. These shown TBNs were measured according to D-44739, 739—because there was little discrimination between used the oils using the D-2896 method. Three of the evaluated detergent technologies are being compared in this evaluation. The TBN depletion rates for a calcium sulfonate and a calcium phenate were found to be similar. A magnesium sulfonate, however, behaved differently; its TBN depletion rate was slower. These findings support work done by other researchers in the GM 6.5L engine test (9). Based on this observation alone, one might conclude that the magnesium sulfonate containing oil has better ESI capabilities than the other two test oils. However, the wear metals analyses show a different picture. The used oil lead content, shown in Figure 2, indicates that the lead corrosion rate is highest for the magnesium sulfonate containing oil.

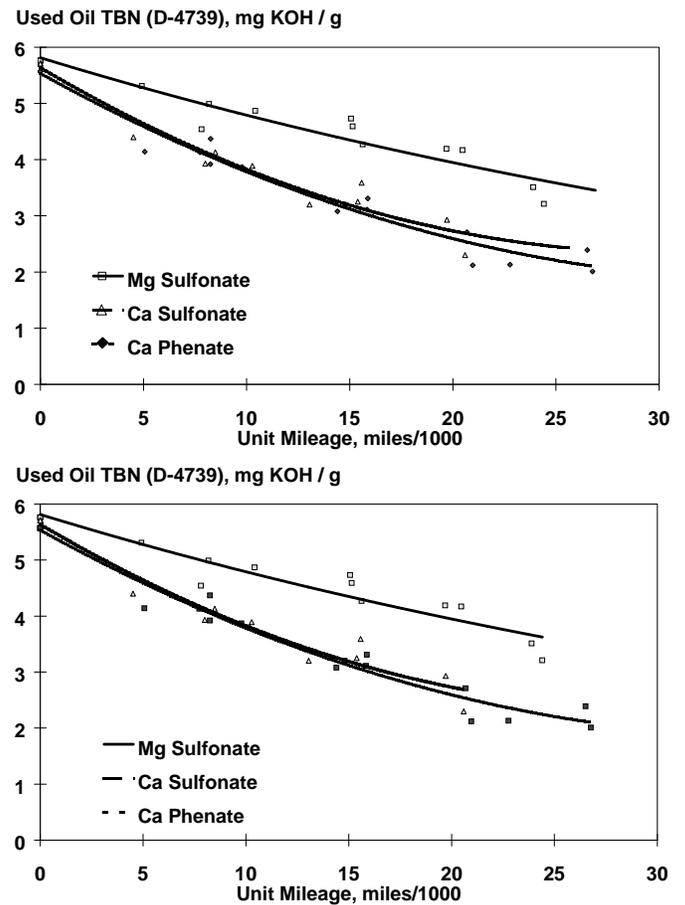
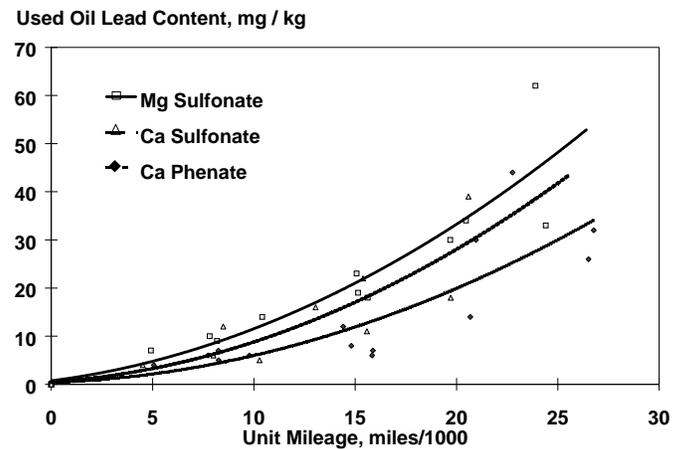


Figure 1 - Field Test Used Oil TBN (D-4739)



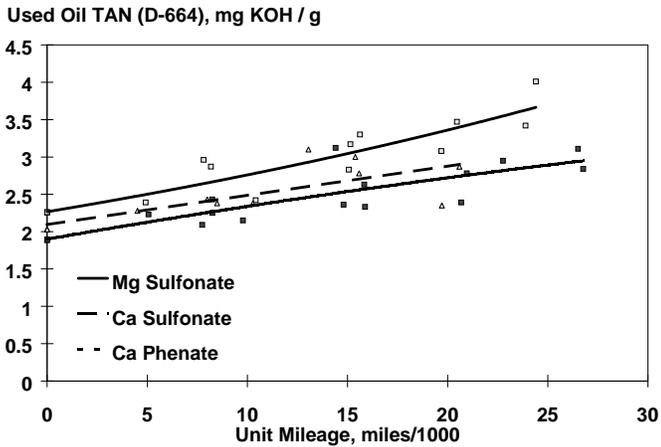
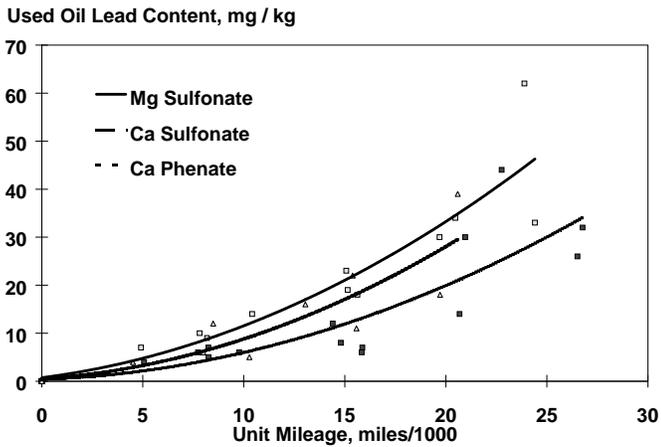


Figure 2 - Field Test Used Oil Lead Content

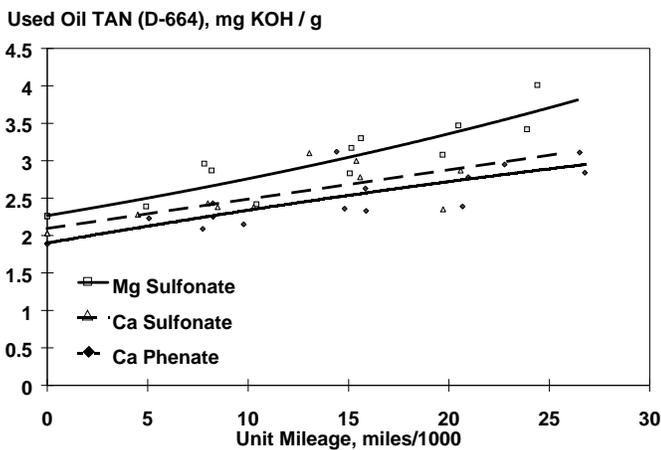


Figure 3 - Field Test Used Oil TAN (D-664)

One explanation for the difference in TAN lead content is that the mMagnesium sSulfonate, possibly being a weaker base in a lubricating oil environment, may not sufficiently neutralize all the acids—, thus reserving base which shows up as a higher TAN. Other researchers have also reported differences in acid neutralization capability for various detergent types (10). If this were true, one would have expected could hypothesize that the use of mthe Magnesium sSulfonate alone containing oil might result in a higher level of corrosive bearing wear. Also it could be a higher expected that the TAN level for observed in the Mmagnesium sSulfonate oil would be expected is higher

than in the Calcium Sulfonate or Calcium Phenate oils. The TAN (D-664) analyses on the used oil samples, shown in Figure 3, support confirm the aforementioned hypothesis. The TAN levels for the mMagnesium measured for the oils containing Calcium based detergents are lower to start with, and increase more rapidly remain lower throughout the drain period.

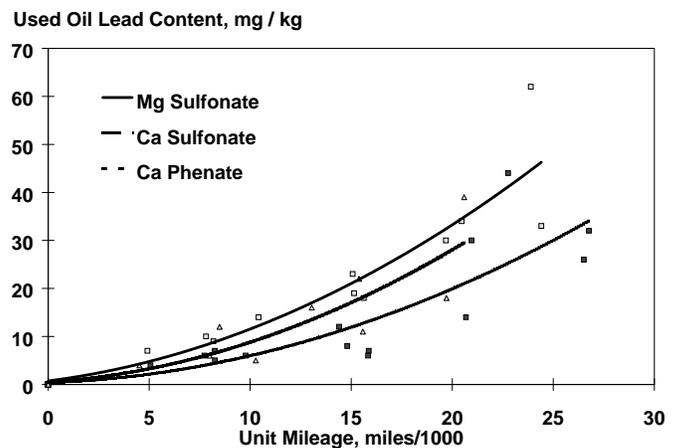
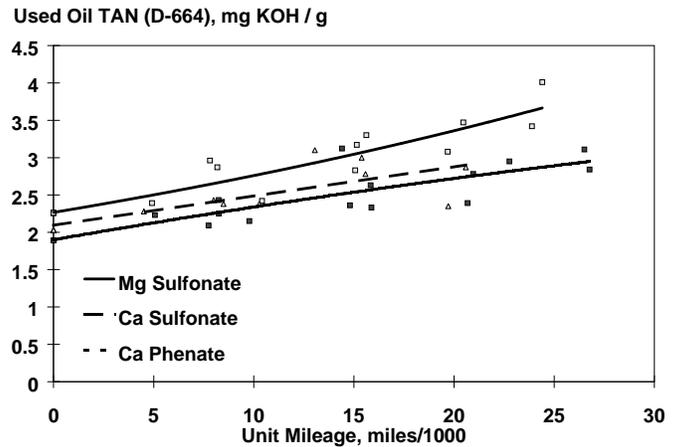


Figure 3 - Field Test Used Oil TAN (D-664)

SUMMARY - It can be concluded that a judgment of the quality of a lubricant, and especially its capabilities for use as an extended service interval oil, cannot be based solely on the TBN measurements. Several other used oil characteristics need to be taken into account to come to the correct conclusions. In the next sections, the results of some bench test work, triggered by our the field test observations, will be discussed.

BENCH TEST OBSERVATIONS

HYPOTHESIS - In the previous section it was suggested that the differences in the used oil lead content, observed in a field test, could be correlated with the differences in TAN increase. At this time, we can only speculate on the reason for these differences in TAN increase however, are not fully understood. One possible scenario, as suggested in the previous

section, is that the mMagnesium sSulfonate does not neutralize weak acids as fully as many acidic species as the cCalcium detergents, therefore it may reserving some of the base, which appears shows up as a higher TBN level. A further, related second scenario is that the base oil, in the presence of mMagnesium sSulfonate alone, oxidizes more rapidly (see below) than in the presence of cCalcium detergents. Oxidation of base oil generates “weak” acids which would show up as TAN if not neutralized. In addition, there is the added possibility that some acids afforded by combustion blowby are similarly weak and not neutralized by mMagnesium sSulfonate., generating organic acids from the hydrocarbon base oil, which show up as TAN, but are not neutralized by the detergent.

Fresh retains of theThe oils that were used in the field test were evaluated in several a number of bench tests to appraise our hypothesis. to find out which of the two mentioned scenarios is closest to reality. The selected bench tests are We chose one an acid neutralization test , and threetwo oxidation tests. for this purpose.

ACID NEUTRALIZATION TEST - The same three field test formulations, identical in every way except for the detergent, were treated with 5 TAN of Section to be written when results are available.oleic acid, a weak organic acid. In addition, the same amount of acid was added to base oil as a reference case. As shown in Figure 4 ____, the magnesium sulfonate did not reduce TAN at all over the base oil case. The greatest reduction in TAN was observed with calcium phenate, but calcium sulfonate also afforded a meaningful reduction in D664 TAN. This is the same ranking as the of TAN increased reduction o observed in the field test.

HIGH TEMPERATURE OXIDATION BENCH TEST - A high temperature oxidation bench test, the Modified IP-48, was run on the three field test oils to examine the impact of oxidation on base depletion in the hotter regions of the engine. This test was run for 4 hours at 200°C in the presence of air but no metal catalyst.

Figure 5 shows the TBN and TAN of these three oils at the end-of-test (EOT). Severe TBN depletion occurred with all three oils, but, like the field test, magnesium sulfonate showed the highest EOT TBN. It also showed the highest EOT TAN, demonstrating again that magnesium sulfonate does not neutralize acidic oxidation products as effectively as calcium phenate.

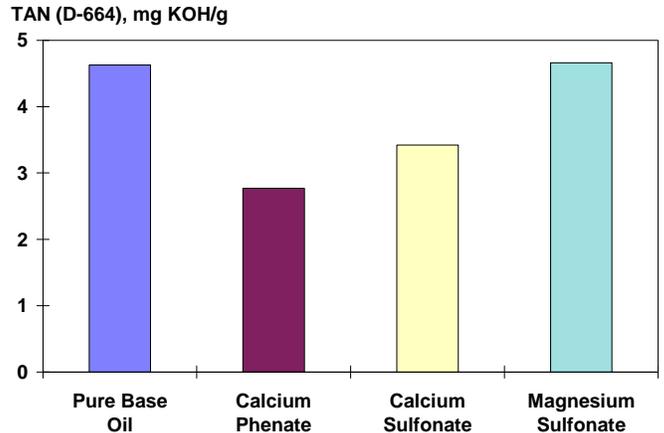
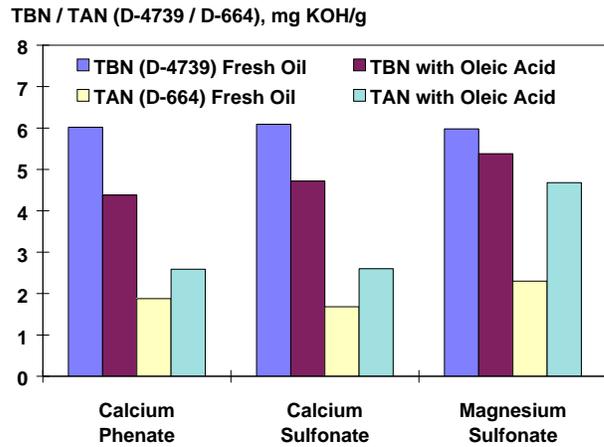


Figure 4X - Acid Neutralization Test TBN Depletion / TAN Increase

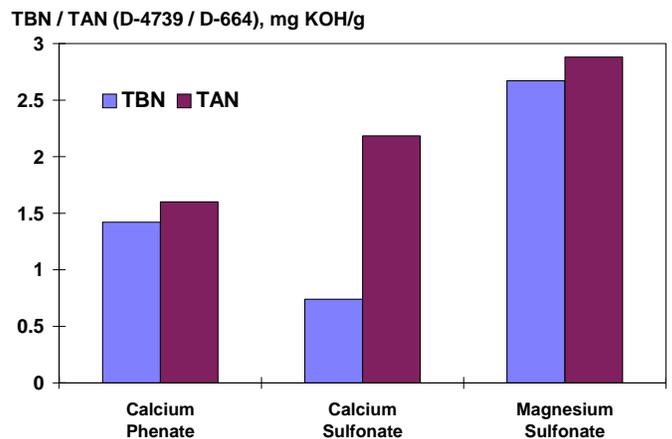


Figure 5 - Modified IP-48 TBN / TAN

HIGH TEMPERATURE OXIDATION BENCH TEST - A high temperature oxidation bench test, the Modified IP-48, was run on the three field test oils to examine the impact of oxidation on base depletion in the hotter regions of the engine. This test was run for 4 hours at 200°C in the presence of air but no metal catalyst.

Figure Y shows the TBN and TAN of these three oils at the end-of-test (EOT). Severe TBN depletion occurred with all three oils, but, like the field test, Mg sulfonate showed the highest EOT TBN. It also showed the

highest EOT TAN, demonstrating again that Mg sulfonate does not neutralize acidic oxidation products as effectively as Ca phenate.

The relative degree of oxidation of the test oils in the Modified MIP-48 bench test is shown in Figure 6. The relative ranking of the calcium phenate and magnesium sulfonate is consistent with the field test data, but the calcium sulfonate, which behaved as good as the calcium phenate in the field, did not look very good in the high temperature MIP-48 test.

OXIDATION BENCH TESTS - Oxidation was also studied in the Indiana Stirring Oxidation Test (ISOT). The results were measured by Fourier Transform Infra-Red (FTIR). The FTIR results of both bench tests and the field test are shown in Figure 6. Although the ISOT reaction temperature (170°C) is higher than the field sump temperature (90 - 110°C), the oxidation levels are in the same order of magnitude, suggesting that the severity of the ISOT is comparable to the field.

PDSC TEST - To supplement the conclusions from the Modified IP-48 and the ISOT bench test, the same field tested oils were evaluated in the Pressurized Differential Scanning Calorimetry (PDSC) bench test. An isothermal test mode (200°C) was used, and the oxygen pressure was set at 100 psi. The induction times are shown in Figure 7. The induction time for the calcium phenate is long, indicating good oxidative stability. The magnesium sulfonate gave a short induction time which indicates poor oxidation stability. This is in line with previous observations from the ISOT, and supports the idea that the magnesium either promotes oxidation, or is not an effective inhibitor. Where the ISOT and the PDSC differ is in the relative ranking of the calcium sulfonate. This might be the result of the difference in temperature at which the oils are evaluated in the two tests.

oxidation bench test. The Indiana Stirring Oxidation Test (ISOT) and the results were measured by Fourier Transform Infra-Red (FTIR). The FTIR results of both the bench and field are shown in figure 4. oxidation results of this bench test, and the oxidation levels found in the field test are shown in figure 4. Although the ISOT reaction temperature (170°C) is higher than the field engine sump temperature (90 - 110°C) in the field test, the oxidation levels are in the same order of magnitude, suggesting that the severity of the ISOT is comparable to the earlier described field test.

Figure -

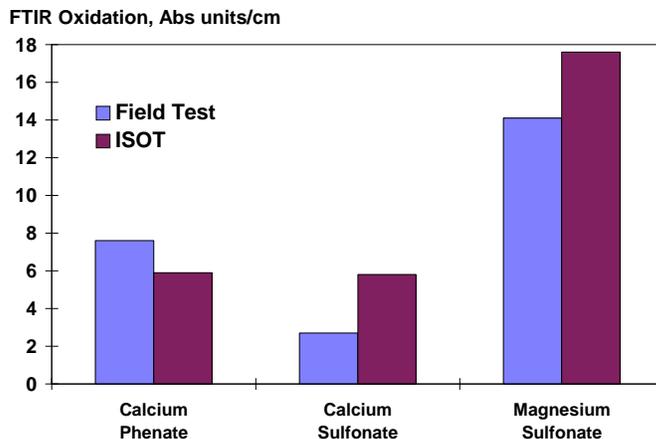


Figure 4 - Field Test versus ISOT Oxidation

Although the correlation between field and bench test is not perfect, both tests indicate that the mMagnesium sSulfonate containing oil gave higher oxidation levels. This observation supports the scenario of increased oxidation of the base oil in the presence of mMagnesium.

PDSC TEST - To supplement the conclusions from the ISOT bench test, mixtures of the same detergents were blended in base oil and evaluated in the Pressurized Differential Scanning Calorimetry (PDSC) bench test; a sample of base oil was submitted for reference. An isothermal test mode (200°C) was used, and the oxygen pressure was set at 100 psi. The measured induction times are shown in figure 5. The induction time found for the mMagnesium sSulfonate is "0," very short, which is in line with previous observations, and supports the idea that the mMagnesium either promotes oxidation, or is not an effective inhibitor.

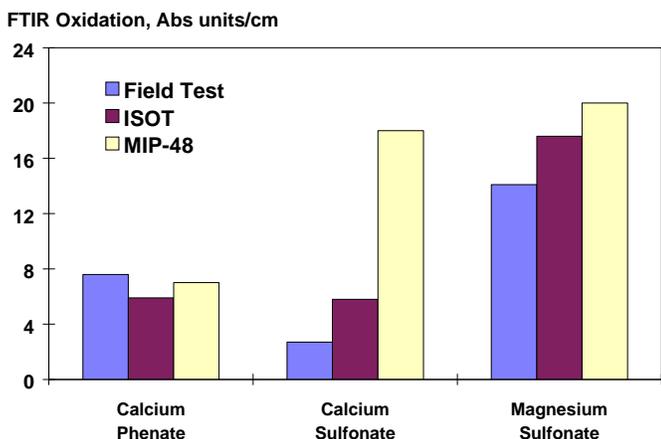


Figure 6 - Field Test versus ISOT Oxidation

OXIDATION BENCH TEST - Oxidation was studied to further the study of the difference in TBN depletion rates of a number of field tested detergents, the oils from the field test were subjected to an

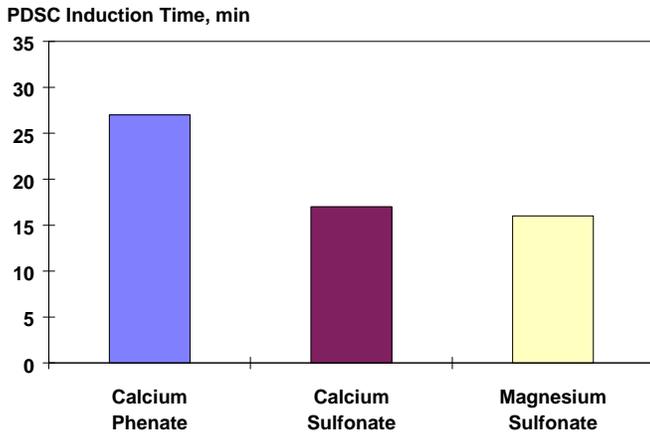
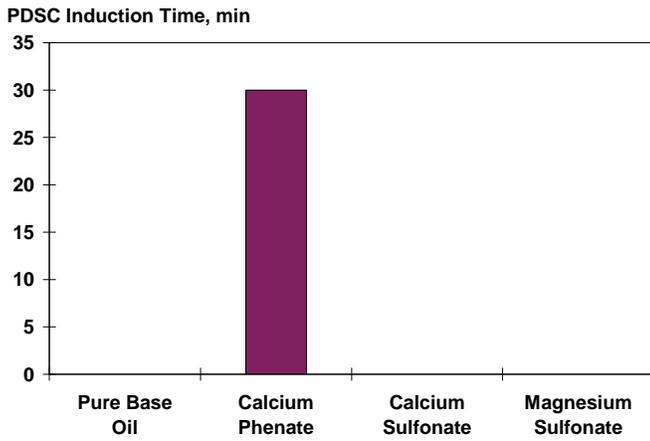


Figure 75 - PDSC Comparison of Detergents

SUMMARY - Although the correlation between field and bench test is not perfect, both field and bench test data indicate that the magnesium sulfonate-containing oil gave higher oxidation levels, and the calcium phenate-containing oil gave lower oxidation levels. The performance of the calcium sulfonate-containing oil seems to depend on the operating temperature of the test. In the field test (90 - 110°C) and the ISOT (170°C), the calcium sulfonate performs as well as the calcium phenate. But in the Modified MIP-48 (200°C), and the PDSC (200°C), the oxidative stability of the calcium sulfonate is not adequate, and its performance is similar to that of the magnesium sulfonate. In general, the bench test data support the hypothesis that differences exist between detergents in their acid neutralization and antioxidant capacities. The acids originating from either the combustion process, via the blowby, or from the high temperature oxidation of the base oil. In the next section a series of engine test results will be examined to see if the phenomenon observed in the field also occurred in the engine tests that will become part of the future heavy duty diesel engine oil specifications API PC-7.

ENGINE TESTING

We compared our own field results with results from four engine tests that will become part of the future heavy duty diesel engine oil specification API PC-7:

Ideally, the engine tests used for the specification of engine lubricants, should evaluate, part of, the performance of those lubricants in a way that is related to the use of the oils in the field. The available test results from the Mack T-9, the Cummins M11, the Mack T-8, and the Caterpillar 1P were screened for detergent comparisons. Some of our findings are discussed in the following sections. The purpose of this screening was to see if differences in detergent behavior found in the field are reflected in the engine tests.

MACK T-9 TEST RESULTS - The most interesting data came from were offered by the Mack T-9 9, which was designed to be primarily a cylinder liner and piston ring wear test. The Mack T-9, more than any other PC-7 engine test test, has the character of an ES extended service interval test. During the test's 500 hour duration test, most a major part of the TBN is will be depleted. , in some cases the TAN increases, in some cases causing a corresponding increase in, and also the lead content—, most likely from bearing corrosion, increases. Four test oils, listed in Ttable 2, were compared in the Mack T-9. With respect to the cylinder liner wear, there was no discrimination between the oils, but oils B and D did give respectively 35 and 20 % gave the lower piston ring weight loss than oils A and C, which were about equal.

**Table 2
Mack T-9 Test Oils**

Test Oil Description	TBN (D-4739)	Detergent Type
A) Internal Reference Oil	6.08	All Ca
B) Low TBN - All Mg	6.97	All Mg
C) Low TBN - Ca/Mg	6.09	Ca/Mg (3:1)
D) High TBN - Ca/Mg	9.83	Ca/Mg (6:1)

The TBN (D-4739) curves of the intermediate oil samples, shown in Ffigure 86, indicate again that the cCalcium- containing oils have a higher rate of TBN depletion than the all-mMagnesium oil. The end-of-test TBN level of the all-cCalcium internal reference oil dropped to near zero; the low TBN cCalcium/mMagnesium oil dropped to 1.2, whereas the all mMagnesium oil dropped only to a level of 2.2. Clearly, the TBN depletion rates of the Ccalcium-containing oils raised some concern over what might have happened to the bearings. Indeed, the used oil lead content curves, shown in Ffigure 97, demonstrate that the internal reference oil did give the highest lead content; the curve starts increasing after only 200 hours. The second worst however, was not a cCalcium-containing oil, but the all mMagnesium oil. In conclusion, among oils of the same general formulation, (ie, excluding reference) the ranking of oils is again reversed going from the TBN plot to the used oil lead comparison.

TBN (D-4739) curves of the intermediate oil samples, shown in figure 6, indicate that the Calcium detergent

based oils have a higher TBN depletion rate than the Magnesium detergent oil. The end-of-test TBN level of the internal reference oil almost dropped to zero. The low TBN - Calcium/Magnesium oil dropped to 1.2, and the all Magnesium oil dropped to a level of 2.2. Clearly, the TBN depletion rates of the internal reference oil, and the mixed Calcium/Magnesium oils raised some concern over what might have happened to the bearings. The used oil lead content curves, shown in figure 7, demonstrate that the internal reference oil indeed gave the highest lead content, the curve starts increasing after 200 hours into the test. The second worst however, was not the low TBN - mixed Calcium/Magnesium oil, but the all Magnesium oil. This demonstrates that the ranking of these oils has been reversed going from the TBN to the used oil lead content comparison.

Used Oil Lead Content, mg/kg

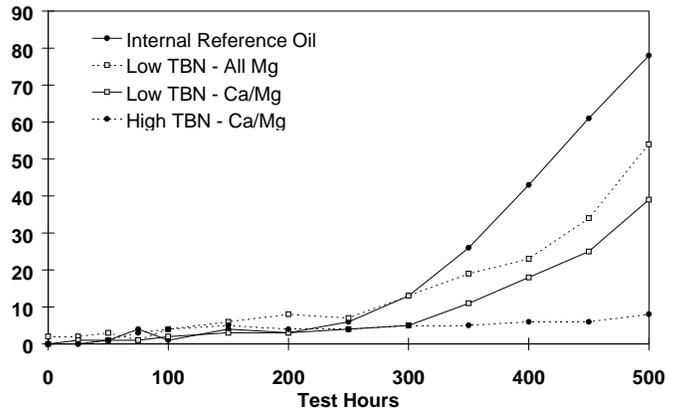


Figure 97 - Mack T-9 Used Oil Lead Content

The TAN increase curves were plotted (Figure 10) in an attempt to understand what caused the lead level to increase for the all-magnesium oil even though its end-of-test TBN level stayed well above 2. As can be seen, the initial TAN for the all magnesium oil was slightly high, but then increased significantly compared to all other oils. This again suggests that acidic species in the oil are not being completely neutralized by the magnesium based detergent. Next the oxidation levels were compared, similar to what was done in the bench test section. These oxidation levels, shown in Figure 11, appear to relate very well with the observed used oil lead contents, lending support to the idea that the oxidation of the base oil created the acids that resulted in both the TAN increase and the lead corrosion of the bearing material.

An interesting observation is that the absolute level of TAN, at which the lead content starts to increase appears to be different for the different detergent chemistries. Based on this observation it is not recommended to set an absolute TAN level as a pass/fail criterion for lubricants, just as no single condemning limit is appropriate for TBN.

Used Oil TAN (D-664), mg KOH/g

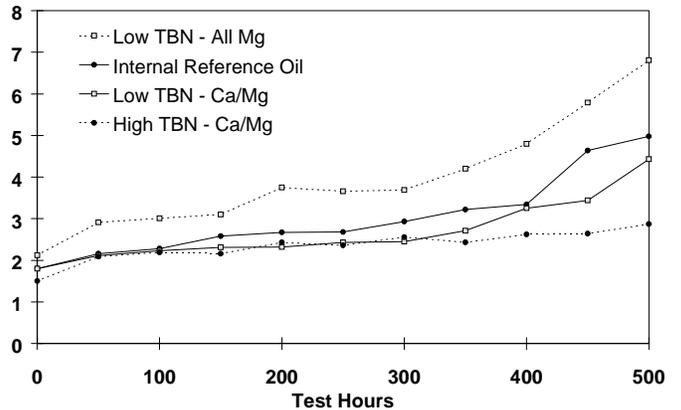
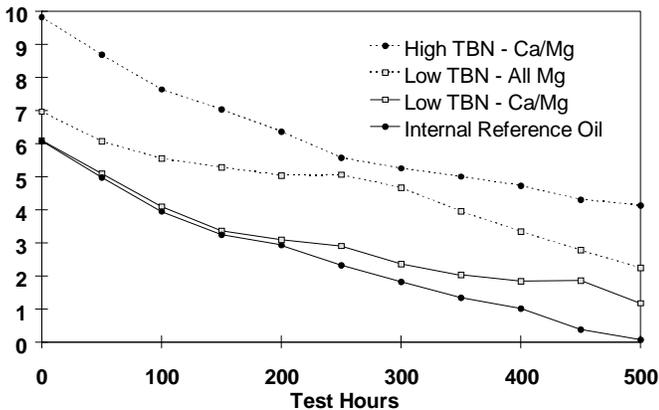


Figure 10 - Mack T-9 Used Oil TAN (D-664)

The TAN increase curves were plotted, (figure 8) in an attempt to understand explain what caused the lead

Used Oil TBN (D-4739), mg KOH/g



Used Oil TBN (D-4739), mg KOH/g

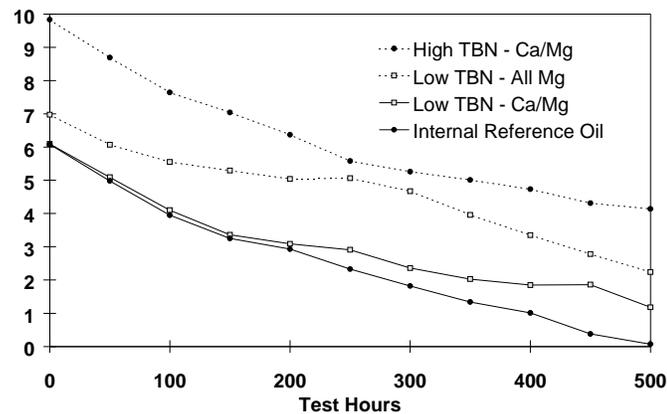


Figure 86 - Mack T-9 Used Oil TBN (D-4739)

level to increase for the all-m Magnesium oil even though, while it's end-of-test TBN level stayed well above 2. As can be seen in figure 8, the initial TAN for the all Mmagnesium oil was slightly higher to start with, but then increased to a significantly higher level compared to all other oils. This again suggests that the acidic species, present in the oil are not being completely neutralized by the mMagnesium based detergent. Next tThe oxidation levels of the oils as a function of the test time were compared, , similar to what was done in the bench test section, to see if the oxidation might have provided the acidic species, present in the oil. These oxidation levels, shown in figure 9, correlated very well with the observed used oil lead contents, lending which lends support to the idea that the oxidation of the base oil created the acids that resulted in both the TAN increase, and the lead corrosion of the bearing material.

An interesting observation is that, for the all-m Magnesium product, the lead content starts to increase when the TAN gets higher than approximately 4. The other oils, which contain more Calcium based detergent, give an increased in lead content when the TAN rises gets above approximately 3. This is a small data set, and until more tests are run, This leads to the conclusion that it it is not recommended to setuse an absolute TAN level as a pass/fail criterion for a lubricants.

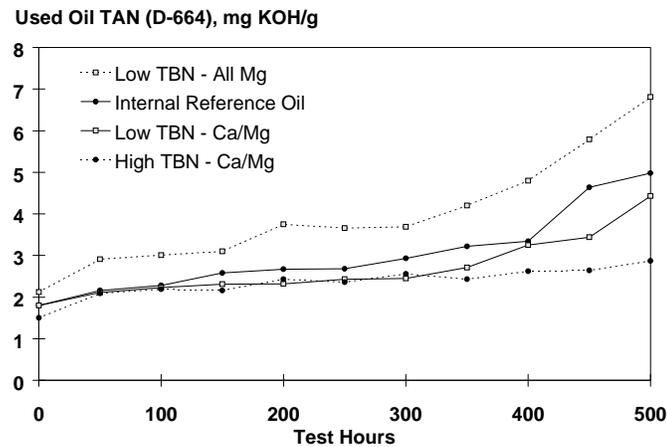


Figure 8 - Mack T-9 Used Oil TAN (D-664)

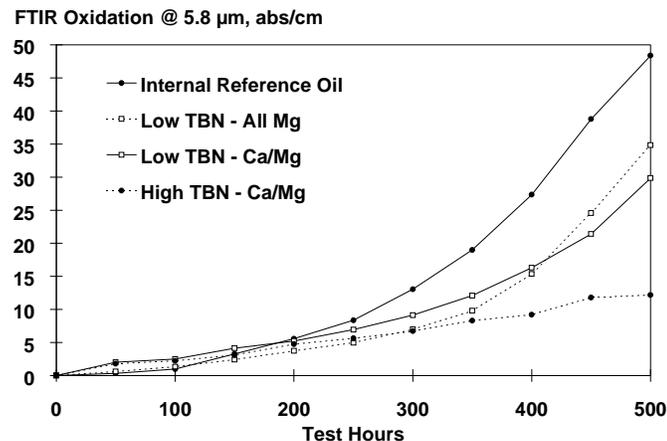


Figure 119 - Mack T-9 End-of-Test Oxidation

MACK T-8 TEST RESULTS - Although the This engine testMack T-8 test does not evaluate engine wear, but it does provide data on TBN and TAN. Figure 120 shows a direct comparison between an all mMagnesium formulation, and an all cCalcium formulation. The baseline of the two formulations was the same and eachthe bars represents the averages of two tests. Again, the cCalcium approach gave a higher TBN depletion, but coupled with a smaller TAN increase. Used oil lead contents in these tests were low in all cases; this is , which is in line with the Mack T-9 tests, where the lead content only started to increase when the TAN exceeded certain levels a level of approximately 4 for an all Magnesium oil, and a level of approximately 2.5 for Calcium based oils (see Ffigures 97 and 108).

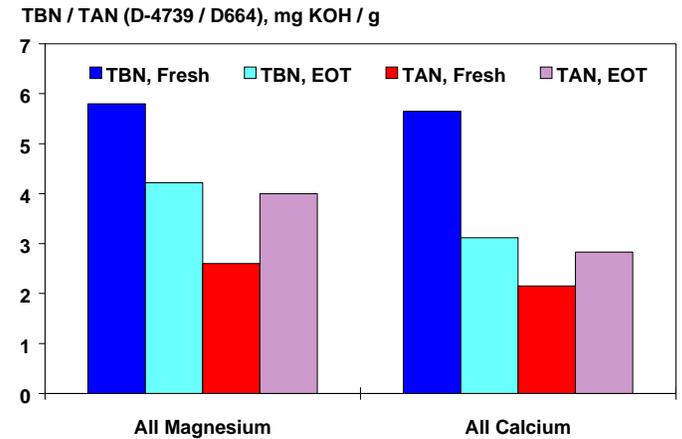


Figure 120 - Mack T-8 Detergent Comparison

CUMMINS M11 TEST RESULTS - A third key test for the API PC-7 specifications is the Cummins M11 high soot test. This test measures sludge formation, oil filter differential pressure increase, and the weight loss fromof the crossheads—, a valve train component. Figure 131 shows a direct comparison between two oils:, Aan all cCalcium, and an all mMagnesium formulation. The baselines of the two oils were identical. The wWear results for the two oils were similar, although there was a directional benefit for mthe Magnesium based formulation. The TBN and TAN data provide the same picture as shown by the Mack T-9 and T-8 data. : The TBN depletion is greaterrate is higher for cthe Calcium based formulation, but the TAN increase is lower. As with the Mack T-8 tests, the TAN did not reach high enough levels to cause any bearing corrosion.

TBN / TAN (D-4739 / D-664), mg KOH/g

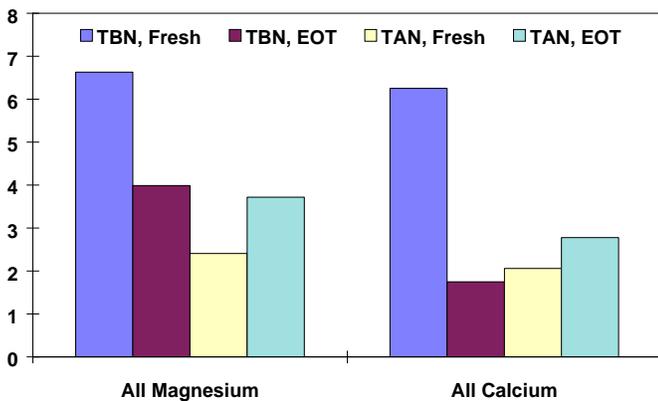


Figure 131 - Cummins M11 Detergent Comparison

SUMMARY - Based on piston ring weight loss data, and TBN depletion rates, one would conclude that a mMagnesium only formulation detergent based oil, and a high TBN oil (Table 2, oils B and D respectively) from table 2) were, were the best performers in the Mack T-9. However, once the used oil lead content, and the TAN increase levels are taken into account, a slightly different conclusion will be drawn. The mMagnesium detergent based oil, although good in preventing ring weight loss, gave higher levels of bearing corrosion. Data from the other PC-7 engine tests, the Mack T-8, and the Cummins M11, support the observations from the Mack T-9 test. Overall, it seems like the best formulating approach for the PC-7 engine tests is a higher TBN mixed Calcium/Magnesium approach, Taking advantage of the strengths, and avoiding the weaknesses of each of the two detergent types.

CONCLUSIONS

One cannot it is impossible to properly characterize a lubricant, and judge it's suitability for extended service intervals use, based on TBN retention measurements alone only. In addition, the ability to control

In addition to TBN, also TAN and especially, but also oxidation need to be evaluated due stability because of the potential impact on , bearing the behavior with respect to corrosion. of bearing materials, , the dispersancy, and perhaps other parameters such as dispersancy need to should be examined. taken into account.

Each of the evaluated detergent types has strengths and weaknesses. , and in order to optimize a lubricant's performance, it may be best to use a well balanced mixture of more than one detergent type The work presented in this paper suggests that some detergents which have good TBN retention, are less effective in neutralizing acids, and may cause lubricants to be less oxidatively stable.

Formulation of a high performance diesel engine oil should be possible using each of the detergent options evaluated in this paper, provided that the other components in the additive package are carefully selected and balanced for optimized performance for PC-7, the best formulating approach utilizes higher TBN and a mixed cCalcium/mMagnesium.

, taking advantage of the strengths of each of them.

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