The Oxidation Stability of Gear Oils in Modern Differentials; A More Hostile Environment: Part Two – Protocol and Precision of the VDOT Gear Oil Test

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SUMMARY

For many different reasons, differential temperatures have increased to levels that may significantly shorten the life of the fluid and threaten the durability of the differential. Automotive and lubricant manufacturers are faced with a serious challenge. They must develop differential fluids with the ability to withstand oxidation with its potentially highly adverse multiple effects. These effects include viscosity increase, deposit formation and oxidative decomposition of the fluid's functionality. Such an effort requires an effective bench screening test because of the time, costs, and limited repeatability associated with fleet tests of differential fluid performance. With this evident challenge, this paper reports development of a test protocol called the VDOT – an acronym for Viscosity, Deposit, and Oxidation Test. This new test measures viscosity and oxidation change as well as deposit formation tendency of gear oils as a means of understanding the relationship between differential temperatures and fluid degradation.

1. INTRODUCTION

The global automotive industry is highly dynamic with constantly improving technology. Some of the many forces creating this drive toward improvement include competition for customer satisfaction at prices the customer is willing to pay, fuel cost savings, and, not least, the global exchange of money and manpower. In the midst of these highly influential factors, a few trends stand out.

Consumer Expectations, OEM Response

Consumer expectations in general drive the industry and new product development. Demand for a variety of vehicle types and configurations have caused OEM's (Original Equipment Manufacturers) to not only ostensibly diversify their product lines while taking advantage of interchangeable systems and components. At the same time OEMs have had to look closer at all vehicle configurations and technologies having impact on use, durability, drivability, and economy of the vehicle. In a quest to develop better, more diverse vehicles that have more interchangeability of parts, no component or development strategy has been left unchecked.

Consumers are demanding better all-weather vehicle handling. In response, automotive OEMs are providing increased performance through a variety of vehicle options, one of which is the use of all-wheel drive.

Over the next few years automakers have announced an impressive number of all-wheel drive product launches In North America alone, it is estimated that the number off all-wheel drive vehicles will rise from the current 3.0% to 8.3% in 2007[1].

Rear Wheel – All-Wheel Drive Trends

Another trend has been an increase in the use of rear wheel drive as opposed to front wheel drive in passenger cars. Advances in technology such as electronic stability protection have improved the on-road handling of the new rear wheel drive vehicles. New, high profile vehicle launches such as the Chrysler 300 and the Cadillac XLR have helped increase the importance of this segment.

The dual trends of all-wheel drive and the resurgence of rear wheel drive have markedly increased the consumer's awareness and expectations regarding the passenger car rear axle and vehicle performance.

Vehicle Performance and Engine Power Trends

Consumers are also demanding increased vehicle performance through increased torque and horsepower and are gravitating to higher displacement engines, often with more cylinders. From luxury cars to economy cars, horsepower is increasing. An example is DaimlerChrysler's Jeep Grand Cherokee offering a 325 horsepower 5.7L V-8 – the largest engine available previously was a 265 HP 4.7L V-8[2]. In most of these vehicle applications the newer high output engines are mated to the same rear axle. Such increase in torque transfer requirements – often on the same axles – are strong tests of differential and fluid capabilities.

Fuel Economy

At the same time that consumers are pressuring industry to increase performance, fuel economy concerns have increased. All of the traditional fuel economy issues remain while new ways to improve fuel economy are identified.

Many engineering attempts to increase fuel efficiency directly or indirectly affect the differential. For example, efforts to reduce air resistance or drag along the underbody of the vehicle also shield the differential from desirable cooling. Similarly, efforts to use more fuel efficient oils often are in the form of lower viscosity fluids and, thus, some viscous protection of the differential bearings is lost. Even technology as fundamental as basic gear design, is being reexamined in an effort to increase fuel economy – design changes that may ultimately stress the differential fluid.

Collective Effects

In summary, all of these trends;

1. the increased importance of rear axle for automotive applications,
2. increased consumer demand for higher output engines, and
3. increased interest in fuel economy,

have created a climate in which not only are the technical demands placed on axles greater, but axle durability and efficiency is expected to be improved.

In other words rear axle applications are facing a "perfect storm" of new challenges. All of these challenges; have generated renewed interest in the optimization of differential design – including the optimization of the differential fluid – to work in conditions considerably more demanding than in decades past.

2. BACKGROUND

This paper is a continuation of work some of which was presented in a previous Colloquium at the Technical Academy of Esslingen in 2004[3]. In that paper experimental design was employed to evaluate the relative effect of time, temperature and iron catalyst (to simulate operating conditions in the differential) on oil oxidation using a modified protocol in the Thin Film Oxygen Uptake Test (TFOUT) apparatus [*].

Influence of Temperature and Time of Exposure

The previous study effectively correlated field and dynamometer experience of some commercially available oils to the modified TFOUT bench test and protocol. The testing also showed the effectiveness of the Taguchi matrix analysis technique in sorting out the influence of several factors on gear oil resistance to oxidation. Results clearly indicated the degree to which temperature, time of exposure, and catalyst concentration has using several axle fluids.

As a consequence of the exponential relationship between oxidation and temperature, it was not surprising to find that operating temperature had the largest effect on gear oil oxidation, followed in severity of effect by the time over which that temperature was applied. Between the limits of 0.5% and 1.5%, the catalyst concentration effect had relatively minor effect and, on this basis, 0.5% concentration of catalyst seemed acceptable.

Variations among Differential Fluids

Again, it was not surprising to find that there were considerable variations among the four gear oils studied regarding their response to the factors of exposure 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.

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

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

3. TEST APPARATUS AND PROTOCOL

The test apparatus was chosen for the advantages of

1. limited sample requirements (1.5 g),
2. relatively short test intervals (60, 90, and 120 minutes), and
3. Comparatively simple catalyst.

Oxidation tests are often imprecise and one of the objectives of this work was to develop a test protocol that would be relatively precise in measuring three important variables simultaneously –

1. oxidation level of the remaining fluid,
2. amount of deposit formed, and
3. Viscosity change in the oil.

The use of three intervals of time exposure to pure oxygen under pressure at 170°C was to provide a measure of the rate of change in the three properties measured.

Test Apparatus

The apparatus used in the oxidation of gear oils in this study is commonly known as the Thin Film Oxidation Uptake Test (TFOUT, ASTM D4742 **[1]**). Information on this instrument and studies conducted with it are available in the literature **[2, 3. 4]**.

Test Protocol

The Viscosity-Deposits-Oxidation Test (VDOT) test protocol was developed after a considerable study of the factors influencing gear oil oxidation and is given in Appendix 1.

4. PRECISION STUDY

In the development of a test protocol it is important to obtain a reasonably high level of precision. Considering the small sample size (which is desirable for reducing the time required to obtain information from the test) and the somewhat difficult-to-control nature of oxidation tests in general, the first part of the application of the new protocol was to determine its precision.

Reference Gear Oil

A well-known gear oil also used in factory-fill service was selected for determining. This is identified in this paper as Oil A, a SAE 75W-140 gear oil.

60-Minute Test Protocol

Results of a series of four analyses of Oil A at 60 minutes using the VDOT protocol are shown in Table 1

Table 1: Test Precision: 60 Minutes

Oil A	Viscosity change	Oxidation (A/cm)	Deposits (mg)
Run 1	16.8%	35	16.2
Run 2	19.8%	34	10.9
Run 3	19.7%	33	14.1
Run 4	16.0%	40	14.7
Average.	18.1%	35.5	14.0
Standard. Dev.	1.7%	2.7	1.9

90-Minute Test Protocol

Similarly, results of four analyses of Oil A at 90 minutes are shown in Table 2 for the VDOT protocol.

Table 2: Test Precision: 90 Minutes

Oil A	Viscosity change	Oxidation (A/cm)	Deposits (mg)
Run 1	24%	85	72.1
Run 2	25%	102	79.0
Run 3	24%	88	71.4
Run 4	26%	95	77.2
Average.	24.9%	92.5	74.9
Standard. Dev.	1.0%	6.6	3.2

120-Minute Test Protocol

Table 3 shows results from the 120-minute analyses.

Table 3: Test Precision: 120 Minutes

Oil A	Viscosity change	Oxidation (A/cm)	Deposits (mg)
Run 1	65%	147	127.2
Run 2	63%	143	127.5
Run 3	72%	154	134.0
Run 4	61%	143	113.5
Average.	65.3%	146.8	125.6
Standard. Dev.	4.3%	4.5	7.5

Overall Test Precision

Table 4 shows the overall test precision.

Table 4: Overall Test Precision

Exposure Time	Viscosity Change (Std. Dev)	Oxidation (A/cm Std Dev)	Deposits (mg Std. Dev)
60 Min	1.7%	2.7	1.9
90 Min	1.0%	6.6	3.2
120 Min	4.3%	4.5	7.5
% Standard Deviation			
60 Min	9.4	7.6	13.6
90 Min	4.0	7.1	4.3
120 Min	6.6	3.1	6.0
Average % Std.Dev.	6.6	5.9	8.0

6. DISCUSSION

Precision

As previously mentioned, oxidation tests (in which a variety of chemical reactions may occur and which must be simulated closely for any degree of precision) are among the more difficult bench tests to develop. In the case of the present protocol, efforts to obtain good precision were compounded by the desire to measure three properties important to the oxidation of gear oils – viscosity change, deposit tendencies and oxidation change.

Table 4 shows the overall test precision obtained in this study of an SAE 75W-140 gear oil as well as the percent Standard Deviation which shows how much the three measures change with the property and the value level of the property at the various exposure times.

It is evident that all three properties have relatively good precisions that are reasonably constant with variation of the property measured. The most challenging property – deposit mass – was surprisingly good at 90 and 120 minutes of exposure.

Another benefit of the accumulation of deposit in the glass beaker used in the protocol was the ability to take colored photographs of the appearance of the deposit and its change in appearance with greater exposure. An example is shown in Appendix 2.

Progressive Change

The reason why this test protocol was generated to utilize three time exposures was to permit determination of the rate of change of each of the three properties with exposure time. It was thought that, although interrelated, any one of the three properties might show advance changes independently of the other two

In fact, this turned out to be the case as is evident in the repeatability study of Oil A presented in this paper.

For the four tests composing the precision study, Figures 1, 2, and 3 shows both the progressive change as well as reflect the levels of precision for the three properties of interest. .

Percent Viscosity Change – Figure 1 is a plot of the percent viscosity change in the generation of the composite data for the study of precision of the VDOT protocol.

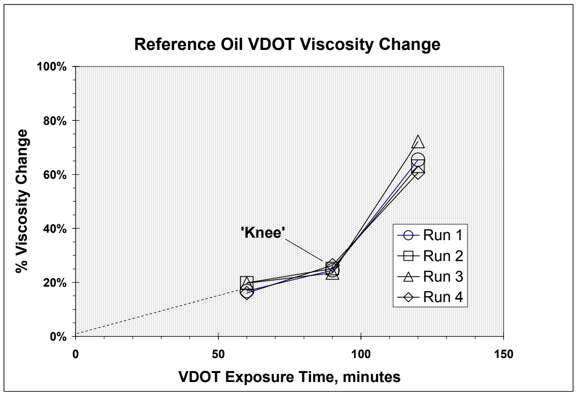

Figure 1 – Plot of the composite percent viscosity change data for Oil A.

A relatively sharp increase in slope of the lines connecting the datum values is identified as the so-called 'knee' of the data. A dashed line is extended to the zero-time point at the test begins and no change in viscosity would occur.

Deposits – A composite plot of the deposits found in the VDOT precision study of the Reference Gear Oil is shown in Figure 2. The data plot as essentially a straight line from 60 minutes exposure and above. Since one would expect no formation of deposits at zero exposure, the deposits shown can be considered to form a 'knee' with regard to the dashed line extended to zero exposure. That is, deposit formation begins at some exposure time less than 60 minutes.

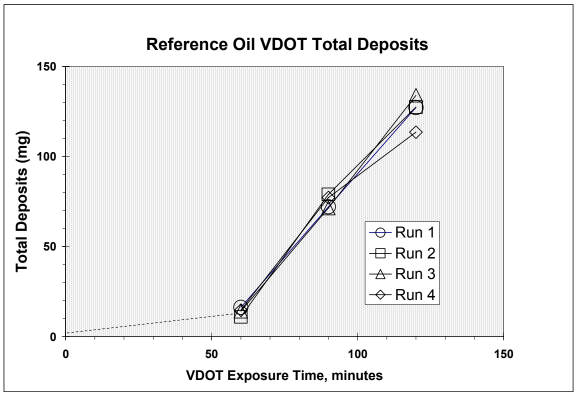

Figure 2 – Plot of the composite deposit data for Oil A.

However, it is interesting to note that deposits increase linearly with exposure time over this range of exposure and it may be likely that a further 'knee' in the curve would develop at some longer exposure interval.

Oxidation – Figure 3 shows the oxidation levels for the composite VDOT data on Oil A.

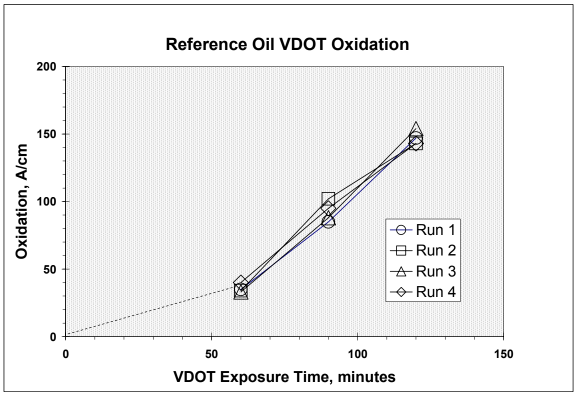

Figure 3 – Plot of the composite oxidation data for Oil A.

Again, the composite data of the VDOT precision analyses plot as straight line. Extension of the oxidation information to zero exposure time (at which there would be no difference in the FTIR spectra and thus zero change in oxidation), again shows that a knee should occur in the oxidation curve.

7. DISCUSSION

As mentioned previously, this study was initiated to develop a repeatable bench test that replicates the oxidative degeneration of gear oils in automotive differentials.

Strategy and Challenges of Test Development

Lubricant and fluid test method development for any application is a matter of first gaining understanding of the requirements of the lubricated and/or hydraulically lubricated mechanism in service. At this point, there is often the opportunity of selecting among a number of possible bench tests which may be applied or modified to give the desired test conditions. In some cases, it is necessary to develop a new bench test to meet the need.

However, this is the stage at which the technical level of understanding of both the mechanism to be emulated and the bench test to be applied is often severely challenged. This is particularly true in the development of oxidation and deposit tests which are a combination of applying both relevant chemistry and physics.

The development of the VDOT test method is a good example of the ease with which knowledgeable assumptions can appear sound but ultimately be inadequate to meet the desired goal. In this case, the primary goal was to collapse the accumulated effects of half a million miles into a test requiring a few hours.

Sample Size – To avoid a prolonged test, test temperature must be high and size of the sample to be exposed to oxidation conditions must be a small as possible. Yet the sample size must be adequate such that after the test, sufficient fluid sample is left to provide test information. Preliminary studies showed that differential fluids were sufficiently resistant to oxidation to allow use of a 1½ gram sample and 170°C. Considerable subsequent work supported this sample size.

Viscometry – With such limited sample size, reasonably precise measurements of viscosity had to be accomplished with only a few drops of sample. This dictated the use of cone-plate viscometry with a heated platen.

However, the need for viscometric information required knowing both the initial and final viscosities. Since a small amount of ferric naphthenate catalyst in mineral spirits solution was added, it was necessary to add a technique to remove the mineral spirits by ambient vacuum before measuring the initial viscosity at 40°C. This required bringing the sample to constant weight.

The final fluid sample was also a challenge in that all of the remaining fluid component had to be removed from the sample both to be sure to have enough sample to measure and to be sure that no fluid was left behind to give false readings regarding deposit weight. This was accomplished by careful syringing of the fluid and solvent washes through a filter from the sample beaker used in the TFOUT instrument. Any increase in filter weight was added to the determined weight of the deposits. Following the careful washings and their collected in the syringe, the solvent was evaporated from the tested sample until constant weight was obtained.

Deposit Measurement – As might be expected, deposit measurement was a considerable challenge. Surprisingly, however, after the protocol was developed, good repeatability has been shown as evident with Oil A. The most critical factor was in thorough washing and careful capture of the particulate matter in the filter during the gathering of the oil remaining after test completion.

Oxidation Determination – Fourier Transform infrared (FTIR) spectrometry was used to permit subtraction analysis of the sample before and after test. These two FTIR analyses were on the sample fluid containing the ferric naphthenate after vacuum removal of volatiles.

The FTIR analyses were performed on ATR (Attenuated Total Reflectance) plates and the small amount of fluid necessary was mostly recovered for viscometry.

Precision

Precision of the resulting VDOT protocol was considered good – particularly for a relatively severe oxidation test using a very small amount of sample.

Oil A was chosen as a reasonably good differential fluid to provide a good basis of determining the repeatability of the protocol developed. It was a fluid originally used widely in field service and a fluid that somewhat preceded the recent high temperature duress applied by the design of very modern automobiles, engines and underbody configurations.

As such, Oil A can be viewed as a transitional differential fluid and, thus, provides a base line against which more modern fluid development can be compared in the VDOT test.

Comparison of the repeatability of the three components of the VDOT protocol are shown in the following graphs.

Viscometric Response – For greater clarity in regard to the precision of the viscosity change portion of the VDOT protocol, Figure 4 shows the four tests at three exposure times. As previously discussed, such viscometric repeatability for very small test samples under oxidizing conditions is infrequent. It will be of high interest to gather further information on other modern forms of differential fluids to determine whether such precision holds or improves.

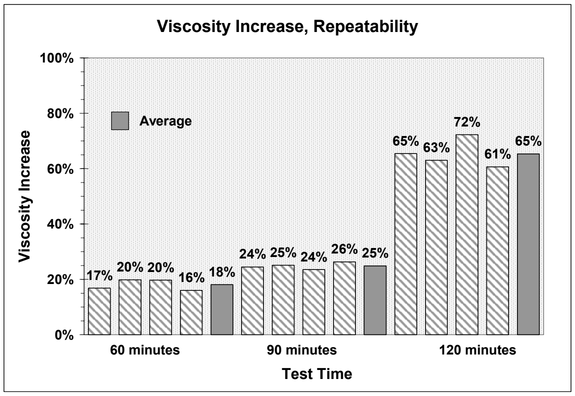

Figure 4 – Repeatability of viscosity change data for Oil A.

At this point it is evident that the VDOT protocol can provide reasonably sharp comparison of this property and its change in the process of oxidative attack on the differential fluid.

Deposit Response – Deposit formation is known to be a difficult property to closely reproduce in oxidation bench tests. Accordingly it is one of the best measures of whether a technique is acceptable. Moreover, the difficulty of obtaining repeatability is one of the main reasons why a number of oxidation tests rely on subjective appearance.

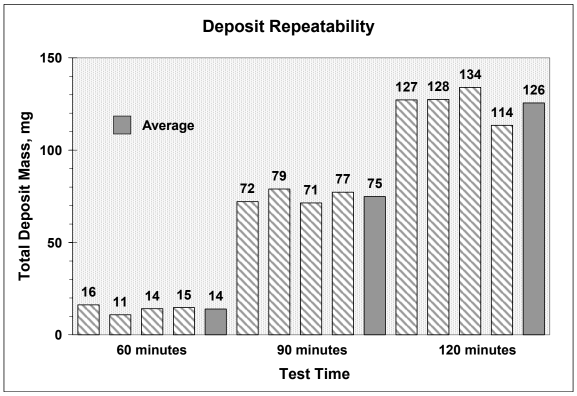

Figure 5 – Repeatability of deposit data for Oil A.

In contrast, Figure 5 and Table 4 indicate that the VDOT protocol does produce reasonably good repeatability at all test intervals. Since deposit formation is one of the more critical aspects of differential fluid decomposition, the evident precision is of significant advantage in the use of the protocol to compare these fluids.

VDOT tests of base oils and additive composition effects on deposits should be interesting and revealing.

Oxidation Response – The cause of changes in viscosity and deposit formation in the VDOT protocol is, of course, oxidation. Choice of the test conditions such as temperature, sample size, catalyst, etc., were all cross-related in the process of developing the protocol.

It was of high interest to measure the degree of oxidation occurring in these tests of repeatability since it would be unlikely that good precision could be generated in viscosity change and deposits without similar repeatability being found in the levels of oxidation at these three intervals of exposure to oxidation conditions.

Figure 6 and Table 4 show the degree of repeatability in the four tests run on Oil A. As expected from the data on viscosity change and deposits, oxidation change is also acceptably repeatable.

8. CONCLUSIONS

A VDOT protocol and first precision results covered in this paper show significant promise in regard to the broad characterization of the response of the differential fluids to increased operating temperatures. As noted, this new test was developed to appraise the rate of oxidation and its effects on two of the important aspects of such degradation in an automotive differential – viscosity change and deposit formation.

The proposed protocol was shown to be surprisingly repeatable for an oxidation test using such small samples under such onerous test conditions. This work represents many trials and tribulations and is the determined result of attention to subtle nuances in observed response with much somewhat iterative small variation in approach. Given the overall precision of this test, it is believed to be acceptable as the first non-subjective bench test and should enable the differentiation of commercial gear oils according to oxidative stability. This test may ultimately be used to help qualify automotive gear oils.

A logical extension of this work would be to:

1. Characterize commercially available differential oils, ranking gear oil according to their oxidative stability.
2. Determine the effects base oils, additives and friction modifiers have on oxidation, identifying key opportunities to maximize oxidative stability while minimizing gear oil cost at a formulation level.
3. Test high mileage axle oils to correlate the oxidative degradation observed in this test to actual oxidative degradation observed in customer vehicles.

10. ACKNOWLEDGEMENT

The authors would like to acknowledge the generous expenditure of time and insightful guidance provided by Christopher Engel and Dennis Florkowski of DaimlerChrysler during the development of this VDOT protocol. Although the Savant Laboratories were responsible for the development of the test method, the background and encouragement of these two individuals were salient to this development of more objective and meaningful differential oxidation test

11. REFERENCES

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APPENDIX 1

This VDOT protocol was developed using the pressure chamber and glass beaker associated with the TFOUT protocol of ASTM D 4742 with several critical modifications.

1. CATALYST

a. Ferric naphthenate is used as the sole component of the catalyst. Its composition and concentration is based on vehicle contamination found in typical differentials vehicles under various service conditions and mileage.

2. SAMPLE PREPARATION

a. Slightly more than sufficient differential fluid sample is taken to give three test portions (~ 5 grams) are placed in a vial.

b. Commercial ferric naphthenate as purchased is added to the differential fluid sample in the vial to give a concentration of 0.5% iron.

c. After thorough mixing by rotating the vial at 30 RPM for ½ hour, the naphthenate solvent is stripped from the 5 g of differential fluid sample under vacuum to bring the sample to a constant mass.

d. The viscosity of the resultant catalyst-containing differential fluid is measured at 40°C. (This mixture will be referred to as 'fresh sample' hereafter in this protocol).

e. Obtain the mass of the special clean, dry glass beaker for VDOT work (see Appendix 2) to within ±0.0002g. Record this weight

f. Place 1.500±0.001g of the oil/catalyst mixture from **Step c** into this special glass beaker and record this mass.

g. Place beaker with sample into a high-pressure reactor vessel.

h. Pressurize with 90 PSI (620 kPa) of 99+% oxygen.

i. Rotate beaker at 100 RPM

3. TEST PROCEDURE

a. Initiate heating to rapidly bring sample to 170°C

b. Heat the first test sample for 60 minutes.

c. At the end of the test cool pressure chamber rapidly as safely possible with care not to spill any of the sample in the beaker within the chamber

d. After the temperature in the pressure chamber is less than 100°C, depressurize slowly to control release of gases from sample and prevent spattering.

e. Remove beaker from reactor carefully, cover with a watch glass or other means and when at room temperature, obtain the mass of the beaker and contents.

f. Subtract mass of beaker and contents after test from initial mass of beaker and contents before test. If the difference in mass is within ±3%, continue the test workup. If the difference is outside of ±3% reinitiate the test.

g. Add 15 mL of a pure, relatively volatile solvent (i.e. heptane) to the beaker containing deposits and oxidized differential fluid and soak 15 hours (overnight) at ambient temperature with watch glass or other form of cover over beaker.

h. Weigh a clean, multilayer filter to within ±0.0002g and record this mass.

i. After 15 hrs of soak, carefully draw liquid contents of beaker through the filter cartridge from **Step h** into a 50-mL syringe using a short length, large diameter (13 gauge) syringe needle.

j. Gently wash contents of glass beaker thrice, each time with 5 mL of same pure solvent and draw off with the same filter cartridge and syringe.

k. Draw three 15 mL of the pure solvent into the syringe to remove any trace of oil from the deposits on the filter and remove filter and needle from syringe

l. Eject contents of syringe into 100 mm diameter Petri dish and place dish into vacuum jar to await the 90-minute and 120-minute samples.

m. Place washed glass beaker under protective cover until 90-minute and 120-minute samples are also prepared for treatment.

n. This protocol from **Step 2e to Step 3l** is then repeated, with additional fresh sample at 90 and 60 minutes, for a total run of three oxidized samples over the different time intervals.

o. At this point remove solvent from all three Petri dishes of **Step l** simultaneously by placing under vacuum for 15 hours (overnight) at ambient temperature. (The oil sample left in the Petri dish at the conclusion of this vacuum treatment will be referred to as 'oxidized oil sample'.)

p. Place the three beakers from **Step m** in a convection oven at ~ 70°C for ½ to 1 hour, remove, let cool to room temperature and weigh to within ±0.0002g and record mass.

q. Draw air by vacuum through filters from **Step k** until constant mass is obtained.

4. DETERMINATION OF TEST RESULTS:

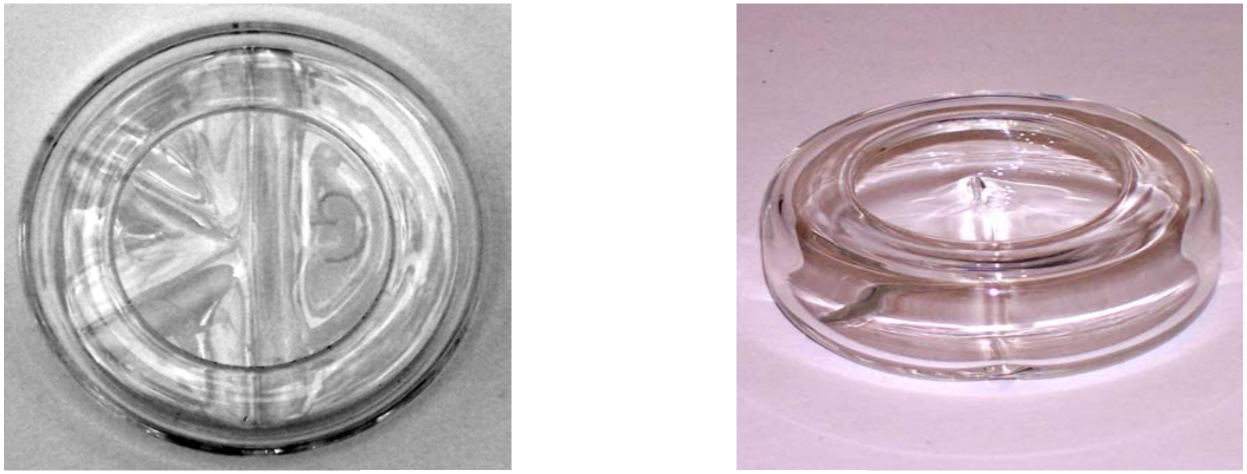
a. Mass of Deposits: The mass of deposits is calculated from the weight difference between the pre- and post-oxidation beaker (**Step 3q minus Step 2e)** plus the mass difference between the initial filter mass and the mass of the filter obtained in **Step 3r**.

b. Percent Change in Viscosity: Determine the viscosity of the oxidized oil sample generated in **Step 3p** again using the cone and plate viscometer applied in **Step 2d** at 40°C.

c. Calculate the percent change in viscosity by subtracting the initial viscosity (**Step 2d)** from the final viscosity (**Step 3p)** divide this value by the initial viscosity and multiply by 100.

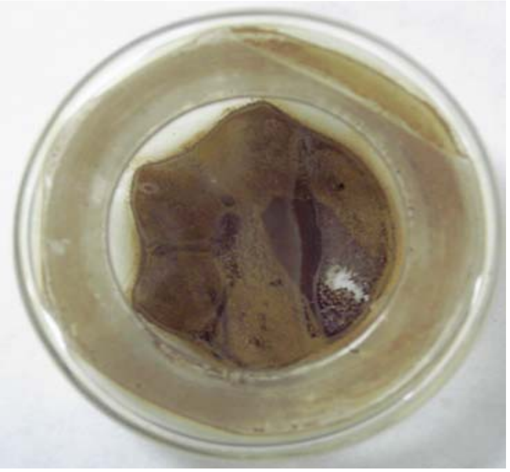
d. Determine the differential oxidation values from FTIR analysis of the sample from **Step 2c** subtracted from the FTIR analysis of the oxidized oil samples from **Step 3p.**

Appendix 2



Clean VDOT Beaker

60 minute



90 minute

120 minute