ASSESSMENT OF USEFUL LIFE OF CURRENT LONG DRAIN AND FUTURE LOW PHOSPHORUS ENGINE OILS

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SUMMARY
Assessment of engine oil useful life is an important step in development of future, low phosphorus, catalyst compatible, and long drain engine oils. This paper describes development and application of a new laboratory screening test, the Ford Oil Aging Test, FOAT, for assessment of engine oil useful life. FOAT simulates the Sequence IIIE engine dynamometer test and evaluates oils on the basis of viscosity increase. It correlates well with both single and double length Sequence IIIE test results. FOAT allows for inexpensive screening of candidate oils prior to engine tests and is currently being used in evaluation of future low phosphorus engine oils. This comparative study has demonstrated that low phosphorus, catalyst compatible engine oils can be formulated to provide similar useful life as current commercial long drain oils.

Keywords: Engine Oil, Oil Aging Test, Oil Useful Life, Emission System Compatibility, Low Phosphorus Oils

1 INTRODUCTION
Next generation engine oils are expected to have improved compatibility with future emission systems while still protecting engines against wear and providing extended oil drain capability [1]. To aid in the development of these oils, it would be helpful to have the ability to assess their performance in the laboratory before subjecting them to extensive vehicle testing. This paper describes development of one such method, the Ford Oil Aging Test, FOAT, that can be used for assessment of the useful life of engine oils. FOAT ranks oils in the same manner as the more expensive and elaborate Sequence IIIE engine dynamometer test. In this work we have utilized FOAT for assessment of the useful life of experimental, emission compatible, low phosphorus engine oils [2]. The oxidative performances of these oils have been compared to that of currently used long drain oils.

2 EXPERIMENTAL
FOAT is performed in a laboratory batch reactor where the oil is aged under simulated engine blowby conditions. The reactor, a model 4560 Mini Reactor from Parr Instrument Company, has a mechanically stirred stainless steel reactor vessel that can hold 100 mL of oil (Figure 1). The gas stream simulating engine blowby was a blend of NO2 and air. The gas mixing and flow rate were controlled by a Model RO-28 control box and Model FC-2900KZ mass flow controllers from Tylan General. The NO2/air primary standard (1000 ppm NO2 in air) used for blending was from Michigan Airgas. During aging the oil samples (2 mL) were periodically withdrawn (at ~24 h intervals) and analyzed for oxidation, nitration and viscosity changes. A Nicolet Avatar 360 FT-IR with OMNIC Integra analysis software was used to measure the oxidation and nitration levels. Oxidation was determined from the peak near 1730 cm-1 and nitration from the peak near 1630 cm-1 in both cases by differential IR using the initial oil as a reference. Kinematic viscosity was measured at 40 °C using Canon-Manning semi-micro calibrated viscometers.

3 TEST DEVELOPMENT
The optimum experimental test conditions were determined using two oils, RO149 and RO152, of substantially different quality. RO149 is a Sequence IIIE borderline pass reference oil (ASTM 1006) and RO152 is a Ford borderline pass double length Sequence IIIE oil meeting Ford specifications [3]. The Sequence IIIE viscosity change results for these two oils are compared in Figure 2. Since oil RO149 is one of the Sequence IIIE reference oils, multiple test results were available for this oil. For oil RO152 only two double length Sequence IIIE test results were available, one being a pass and the other a fail. The test parameters for optimising the test conditions were: concentration of NO2 in the gas stream of 750, 375 and 100 ppm, reactor temperatures of 160 and 170 °C and concentration of an iron oxidation catalyst of 0 and 100 ppm Fe.
3.1 Effects of NO₂ Concentration

The effects of NO₂ concentration in the gas stream on viscosity change during aging of RO149 and RO152 were evaluated using NO₂ concentrations of 750, 375 and 100 ppm. Results of this testing at 160 °C are shown in Figure 3. With 750 ppm NO₂ (solid symbols in Figure 3) there is very little differentiation between these two oils. When the concentration of NO₂ is reduced to 375 ppm (half filled symbols) the differentiation is considerably improved. Thus, it can be concluded that the difference in viscosity change is greater the lower the concentration of NO₂ in the gas. However, reducing the NO₂ concentration to 100 ppm (open diamond for RO149) proved to be impractical due to the increased test time.

The effects of change in NO₂ concentration on the oxidation levels (expressed as carbonyl absorption near 1730 cm⁻¹) are shown in Figure 4. The changes in oxidation levels are directionally proportional to the changes in NO₂ concentration as might be expected since NO₂ is the main initiating free radical in the system. However, the observed changes in oxidation level are quite different between the two oils. With RO149 the oxidation level increases rapidly after a short period at a low rate of change at the beginning of the test. With RO152 change in oxidation level remains at a lower rate for a longer time until it reaches a point where it accelerates and then increases nearly linearly at a rate slightly higher than that observed for RO149. At the time when viscosity starts to increase (Figure 3) there is no significant change in the rate of change of oxidation level (asterisks in Figure 4). The oxidation level at these times is also different in the two oils and is dependent on the NO₂ concentration.
The effects of change in NO₂ concentration on the nitration levels (expressed as nitrate ester, RONO₂, absorption near 1630 cm⁻¹) are shown in Figure 5. For nitration the curves are more complex than they were for oxidation which reflects the fact that the nitrate esters that are being formed are thermally unstable and what is being observed is the net effect of formation and decomposition of these compounds. It is interesting to note that for RO149 there is some correlation between the time when viscosity starts to increase and an increased rise in rate of change of the nitration curves. For RO152 this behaviour is much less pronounced and quite imaginary.

3.2 Effect of Temperature

The effects of increasing the test temperature from 160 to 170 °C on viscosity change are shown in Figure 6.

The good differentiation observed with 375 ppm NO₂ at 160 °C (solid thick lines in Figure 6) was not further improved by going to 170 °C even with the slightly lower NO₂ concentration (300 ppm) that would be also expected to improve differentiation. Thus, the increase in temperature from 160 to 170 °C does not substantially help the differentiation.

3.3 Effect of an Oxidation Catalyst

The effect of an oxidation catalyst on viscosity change was assessed by adding ferric octoate (100 ppm Fe) to RO149 and then aging the oil at 160 °C with 750 and 100 ppm NO₂. At 750 ppm NO₂ the effect of the oxidation catalyst on viscosity change was minimal (filled squares in Figure 7). However, at 100 ppm NO₂ the oxidation catalyst has a significant effect (filled diamonds) on viscosity change. Thus, at lower concentrations of NO₂, the oxidation catalyst reduces the time to viscosity increase.

3.4 Final Test Conditions for FOAT

The final test conditions chosen based on the above test results are listed in Figure 8.

The temperature of 160 °C was chosen since the higher temperature (170 °C) did not improve the separation. The NO₂ concentration in the gas stream was chosen to be 300 ppm as a compromise since higher levels of NO₂ are detrimental to discrimination and lower levels result in test times that are impractically long. Typically, in IIIE tests the Fe concentration gradually increases during the test due to wear and the final concentration of Fe is 100 ppm. Since in FOAT Fe is added at the start of test and no wear iron is generated in the test an amount of Fe half of that expected in the IIIE test was used to represent an average value.

3.5 Correlation of FOAT with Sequence IIIE

Using these final test conditions, the FOAT viscosity changes for oils RO149 and RO152 are compared in Figure 9. The separation between the oils is within the boundaries of the IIIE test in which significant run to run variation is observed. This should be much less of a problem in the laboratory FOAT.
3.6 FOAT Repeatability

The repeatability of FOAT was determined for two tests on RO152 and one test on RO168, which is a new blend of RO152. The results of these tests are shown in Figure 10. Test #1 on RO152 and the test on RO168 give identical results. Test #2 on RO152 gave slightly different results but the variability is much less than it was in the engine test. The range of times to 200 percent change in viscosity is 4.6 h, which is substantially less than that for Sequence IIIE (approximately 30 h).

3.7 Long Drain Oils

FOAT results for three current commercial, long drain oils (0.10% P) (RO152, RO160 and RO183) are shown in Figure 11a. All these oils meet Ford Double IIIE requirements.

3.8 Low Phosphorus Oils

FOAT results for four 0.05% P oils (Oils E-I) from different suppliers are compared to those obtained with long drain oils in Figure 11b. The results show that low phosphorus oils can be formulated to meet double Sequence IIIE requirements. Three out of four oils met the requirement.

4 CONCLUSIONS

A Ford Oil Aging Test (FOAT) for assessment of the useful life of engine oils has been developed. A comparison of viscosity changes occurring in FOAT with those occurring in the Sequence IIIIE engine test showed that these tests directionally correlate. It was demonstrated that the FOAT can be used for predicting oil performance in Sequence IIIIE and double length Sequence IIIE tests. FOAT results indicate that low phosphorus, catalyst compatible engine oils can be formulated to provide similar useful life as some current commercial long drain oils.

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6 REFERENCES