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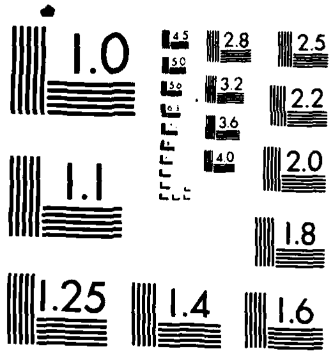

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Bethesda, Maryland 20084

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PREDICTIVE TEST DEVELOPMENT FOR NAVY DIESEL ENGINE LUBRICATING OILS - FINAL REPORT

by

M. D. Klinkhammer

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SHIP MATERIALS ENGINEERING DEPARTMENT

RESEARCH AND DEVELOPMENT REPORT

November 1985

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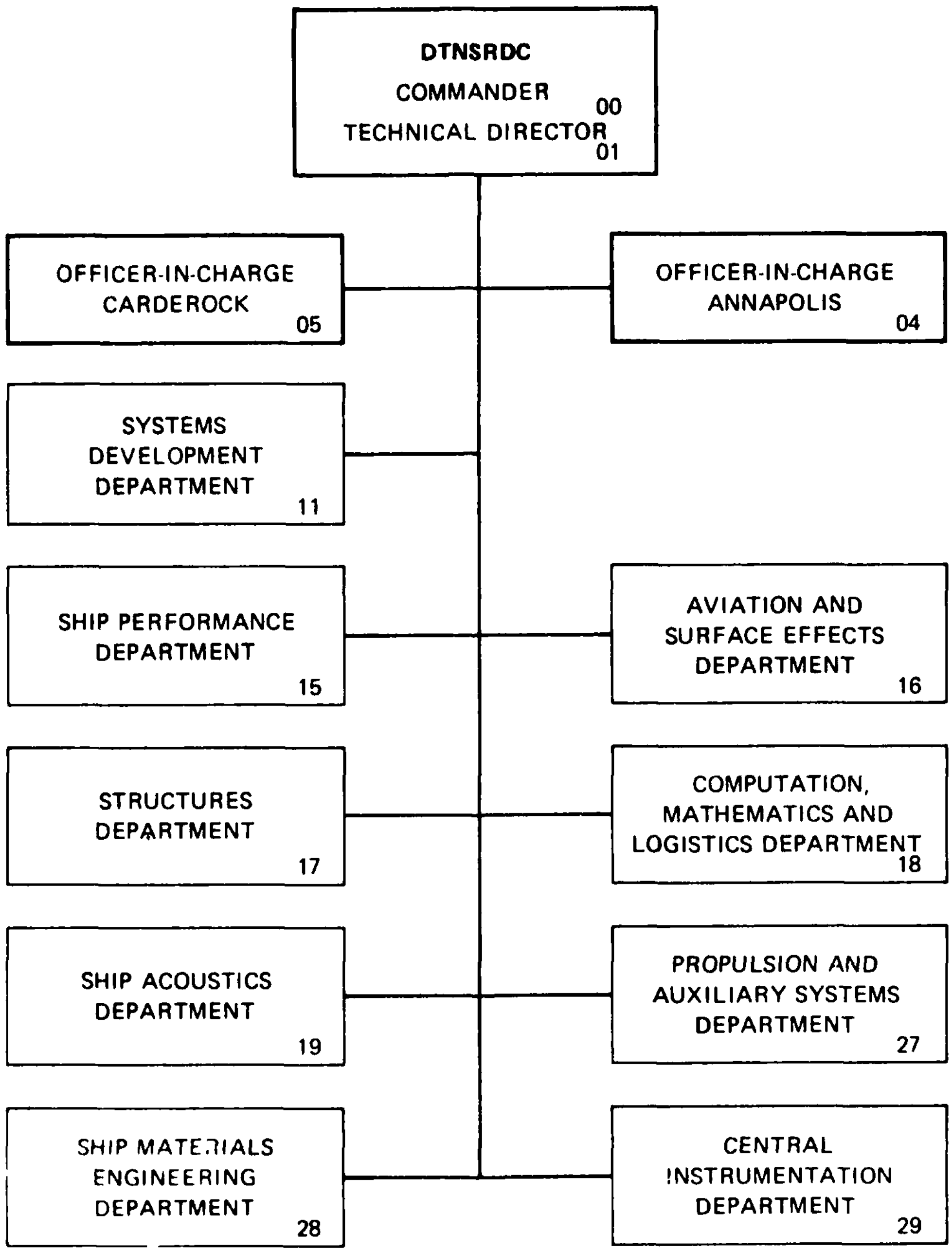

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The objective of this project was to reduce the time and expense of qualification testing for candidate MIL-L-9000 diesel engine lubricating oils. Work was focused primarily on finding a short term test to correlate with Caterpillar 1G2 engine test deposit ratings. Because the 1G2 engine test deposit ratings have poor repeatability, bench test correlations were attempted using only reference oils, i.e., those oils having a large number of engine test results.

Three bench tests were evaluated: the Alcor 1G Deposition Test, a pressure differential scanning calorimetry method, and a microoxidation method. The Alcor Test gave good predictions of weighted total demerits by Caterpillar 1G2 for the seven reference oils available. The pressure differential scanning calorimetry method also gave a good correlation with

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1G2 but only five reference oils were available for evaluation. The microoxidation method shows promise but needs more development. These tests should be evaluated further as additional 1G2 reference oils become available.

We recommend that the Alcor 1G Deposition Test should be considered as a replacement for the Caterpillar 1G2 engine test requirement when the present qualification procedures do not provide sufficient MIL-L-9000 oils and when full requalification testing is not considered necessary due to minor formulation changes.

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LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
°C	Degrees Celsius
HPLC	High performance liquid chromatography
GPC	Gel permeation chromatography
GM	General Motors
l	Liters
MPa	MegaPascals
ml/min	Milliliters per minute
NBS	National Bureau of Standards
lb/in^2	Pounds per square inch
PDSC	Pressure differential scanning calorimetry
RBOT	Rotating bomb oxidation test (ASTM Method D2272)
TGF	Top groove fill
UV	Ultraviolet
WTD	Weighted total demerit

ABSTRACT

The objective of this project was to reduce the time and expense of qualification testing for candidate MIL-L-9000 diesel engine lubricating oils. Work was focused primarily on finding a short term test to correlate with Caterpillar 1G2 engine test deposit ratings. Because the 1G2 engine test deposit ratings have poor repeatability, bench test correlations were attempted using only reference oils, i.e., those oils having a large number of engine test results.

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We recommend that the Alcor 1G Deposition Test should be considered as a replacement for the Caterpillar 1G2 engine test requirement when the present qualification procedures do not provide sufficient MIL-L-9000 oils and when full requalification testing is not considered necesary due to minor formulation changes.

ADMINISTRATIVE INFORMATION

This project was funded by Naval Sea Systems Command (SEA 05R25) through the Marine Tribology Block, Program Element 62761N. Work was performed under DTNSRDC Work Units 2832-103-42, 2832-101-42, and 2832-100-42. This final report met Milestone C2(a)6 of the Marine Tribology Block Plan.

INTRODUCTION

The U.S. Navy must continue to obtain good quality oils to maintain diesel engine lubricating oil life and keep engine maintenance requirements at current levels. Diesel lubricating oil quality is assured by qualification testing which includes using actual diesel engines as test equipment. Candidate diesel engine lubricating oils must pass the GM3-71 and Caterpillar 1G2 engine tests to qualify as MIL-L-9000G[1]* oils. These engine tests are lengthy and costly and change in oil formulation requires requalification. In the past excessive qualification testing was not a problem because oil suppliers were able to draw upon constant base stock sources. Recently, suppliers have been forced to obtain crudes from varied sources with resultant more frequent base stock changes. Although the current availability of MIL-L-9000 oils is satisfactory, this was not the case in recent years when the number of qualified products fell to undesirable levels. Several approaches were taken to improve the situation. Commercial oils were evaluated as MIL-L-9000 substitutes and some candidate MIL-L-9000 oils were qualification-tested at government expense under NAVSEA-sponsored projects. The approach taken in this work has been to try to simplify qualification procedures and encourage more suppliers to submit their oils for qualification testing.

Again, the most lengthy and costly MIL-L-9000 qualification requirements are the two engine tests. Our goal has been to develop methods or tests which could replace one or both of these engine tests while still assuring that qualified oils give good service.

RESULTS AND DISCUSSION

SURVEY OF INDUSTRY AND GOVERNMENT

One of the first tasks attempted was to determine what screening tests and analytical methods were being used by industry, government laboratories, and the military to evaluate diesel engine oils. Also, we sought opinions on whether engine test requirements are needed in MIL-L-9000 and which engine test should be retained if only one was required. Detailed results of this survey were reported previously; our findings are summarized below.

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*A complete list of references used appears on page 15.

Industry takes the position that engine tests are needed to ensure that diesel lubricating oils will perform adequately in service. To that end, much work has been done to attempt to correlate bench tests with engine tests, and several are used to screen oil formulations prior to engine testing. There was a reluctance to discuss their screening tests, many of which they consider proprietary. However, some of these screening tests were discussed in a presentation by Asseff[2] of the Lubrizol Corporation at a 1977 recycled oil conference. Panel-coker tests are often used to simulate deposits produced in diesel engine operation. The National Bureau of Standards (NBS)* and military laboratories were more optimistic about the possibility of being able to predict the engine performance of an oil by short-term tests.

Although some industry representatives were more familiar with the Caterpillar 1G2 engine test, the GM3-71 was considered more important for MIL-L-9000 qualification because the presence of seawater and silver bearings in the GM3-71 test makes it more comparable to Navy diesel engine service conditions. They suggested that if one of the engine tests were to be removed it should be the 1G2.

U.S. industry representatives to the Navy Quadrapartite meetings also made suggestions for improving diesel oil availability to the Navy. One suggested two specifications - one oil for EMD-type engines and another for GM3-71-type engines. However, this is contrary to the attempts of the U.S. Navy to reduce the number of lubricants needed for Navy ships. Another suggested the use of the Army diesel lube, MIL-L-2104C,[3] as an emergency substitute oil or even as a normal procurement. We have since tested two MIL-L-2104C oils against MIL-L-9000 requirements; both failed the engine test requirements. The Caterpillar 1G2 is run under more severe conditions (1% sulfur fuel and no 120-hour oil drains) for MIL-L-9000 qualifications than the standard 1G2 test as run for MIL-L-2104C qualifications. Also, seawater, of little concern to Army diesels, is added to the GM3-71 test oil for MIL-9000 qualifications.

*Definitions of abbreviations used appear on page iv.

Changes in procurement procedures for Navy diesel oils may be necessary as future Navy diesel engines and fuels place greater demands on the lubricating oil. The Navy should evaluate developments and specifications of commercial railroad and marine diesel lubricants. The railroad industry uses similar engines (including silver-lined bearings) to those used by the Navy, though it does not have the sea-water capability concern.

Other Navies were found to be using a number of engine tests besides the GM3-71 and the Caterpillar 1G2. They have used the Rootes TS3, Petter AV1, Petter AVB, and Petter W-1.[4] The Admiralty Oil Laboratory has used the Petter W-1 gasoline engine as a relatively cheap oil oxidation stability test to determine the effect of base oil or additive changes in previously qualified products.

The U.S. Army also has had an interest in methods other than engine tests for assessing oil quality. Southwest Research Institute, under contract to the Army, developed a bench deposition test, called LUBTOT,[5] based on a modification of the Alcor Test Fuel Thermal Oxidation Tester. Although they considered the test effective in evaluating deposition tendencies of oils the results did not correlate with the Caterpillar 1G2 test.

The NBS has reviewed several bench tests for assessing automotive crankcase oils[6] and an NBS-developed bench test for assessing automotive lubricants is being evaluated in ASTM. This test[7] is a thin film oxygen uptake method based on a modification of the standard rotary bomb oxidation test apparatus. A catalyst mixture of oxidized fuel and metals is used to oxidize the oil. A different catalyst mixture would need to be developed if the test is to be useful for diesel lubricant evaluation.

The ultimate goal of this project is the elimination of both engine tests from MIL-L-9000 requirements. Our survey indicated, however, that the best approach would be to first try eliminating one engine test and consequently have spent most of our effort trying to develop short-term tests that correlate with the Caterpillar 1G2 engine test so that engine tests can be "retired." There are two reasons for this choice: the GM3-71 is more representative of Navy service requirements (and therefore is preferred over 1G2 for retention) and the pass/fail criteria are more numerous for the GM test unlike the 1G2 which has only top groove fill (TGF), weighted total demerit (WTD), piston skirt cleanliness, and bearing weight loss requirements. In addition, it is unusual for an oil to pass the GM3-71 requirements and fail Caterpillar 1G2 requirements. We decided that even if the correlations developed were insufficient to warrant replacement of one of the engine tests, a short-term test would still be useful to the Navy. For example, if only minor formulation changes are made to a qualified product full engine qualification testing may not be required if a short term test shows no adverse change.

BENCH TEST DEVELOPMENT

Modified RBOT Tests

The Center tested diesel engine oils using a modified ASTM D2272, "Oxidation of Steam Turbine Oils by Rotating Bomb (RBOT)," in 1975. Five oils were tested at 121°C and an apparent correlation of bomb life in minutes with viscosity increase in the GM3-71 engine test was found. We evaluated this method further by testing an additional five diesel oils. The results did not warrant further evaluation of this test. Ku and Hsu[7] have more recently reported on a correlation of bomb life by a modifed RBOT, with viscosity increase in the ASTM sequence IIID engine test for gasoline engine lubricating oils. Their modifications included: a higher temperature (160°C), a catalyst mixture of oxidized fuel and metals, and a change in test beaker design to produce a thin film of oil. This test could probably be made useful for diesel engine lubricating oils by using a different catalyst mixture to simulate diesel engine conditions.

Alcor 1G Deposition Test

Alcor Inc. developed a bench test to correlate with weighted total demerits from the Caterpillar 1G engine test. The Alcor 1G Deposition Test circulates preheated oil (1.25 liters at 149°C) together with nitrogen and water-saturated air through a heater tube. The heater tube has a temperature gradient of 304°-382°C. The oil is recycled for

48 hours or until sufficient heater tube deposits form to raise the maximum heater temperature to 538°C. A visual rating of the tube is made and the tube deposits are weighed. These two factors are combined to give a predicted Cat 1G WTD value.

Our evaluation of this test showed a good correlation with Caterpillar 1G and 1G2 engine WTD values (1G2, a revision of 1G, uses a different piston). This correlation is demonstrated in Table 1 and Figure 1. Although other oils were tested by the Alcor test, those oils had only a single Cat engine test result. As noted in Table 1, the engine WTD values have poor repeatability. Therefore, only reference oils, i.e., those with several engine test results, are useful in assessing correlations with bench tests. As mentioned above, the MIL-L-9000 1G2 engine test is more severe than the standard 1G2 test used with these reference oils. MIL-L-9000 qualification requires the use of 1% sulfur fuel and no oil changes versus 0.37-0.43% sulfur fuel and 120 hour oil changes for the standard 1G2 test.

TABLE 1 - ENGINE WTD VS. ALCOR PREDICTED WTD FOR REFERENCE OILS

Oil No.	Engine WTD	No. of Engine Tests	Alcor Predicted 1G WTD	No. of Alcor Tests
185	121.9 +47.4*	9	126.7 +5.0	3
CCL-L-738	349.0 +184.1*	9	349.2 +16.0	5
800	136.4 +48.4*	62	128	1
801	337.9 +104.3**	43	260	1
802	182.2 +57.8**	272	172.6 +8.3	5
803	394.9 +112.4**	110	>400	1
803-1	348.2 +81.0**	105	390	1

*Weighted total demerit plus or minus standard deviation for 1G engine test.

**Weighted total demerit plus or minus standard deviation for 1G-2 engine test.

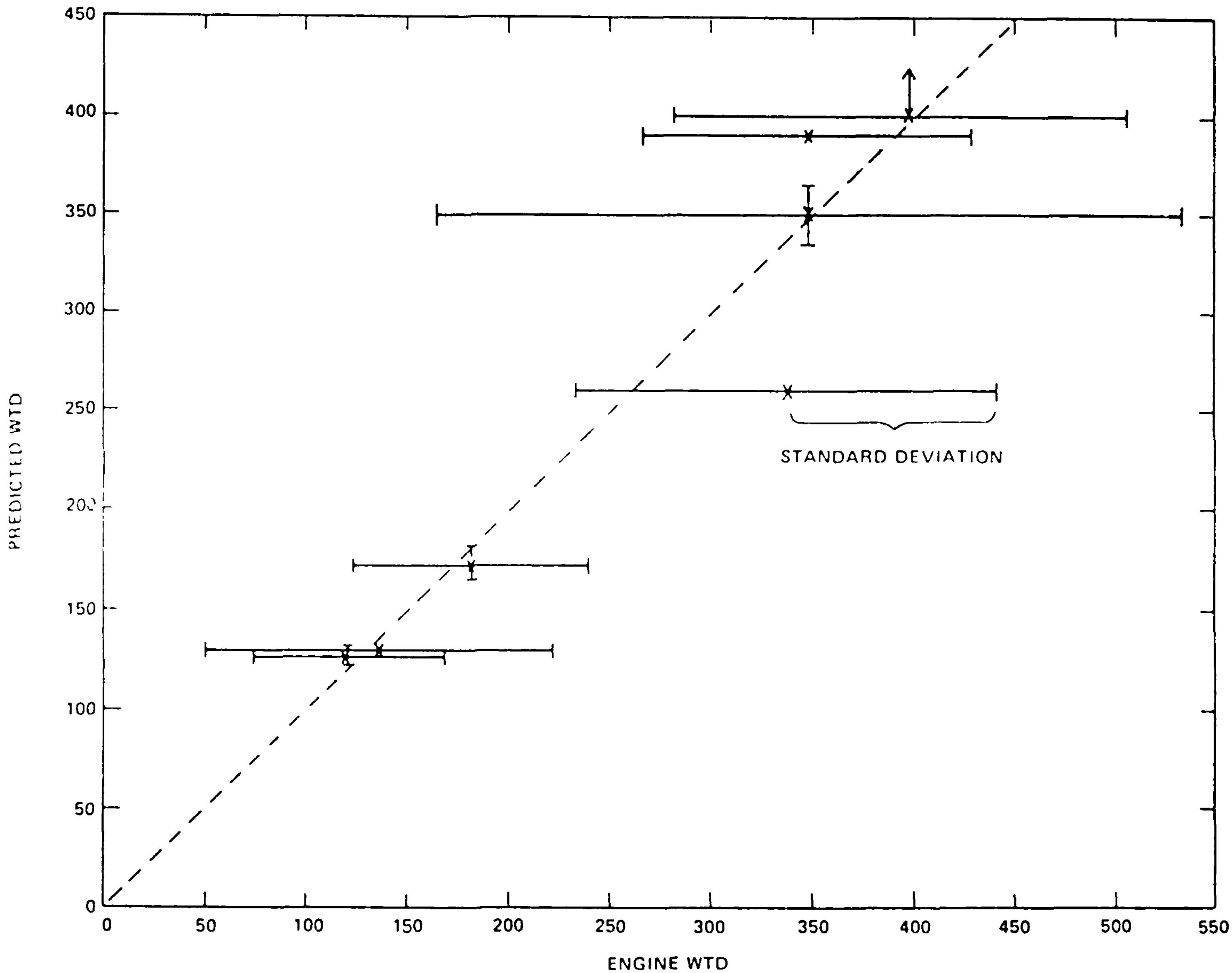

Figure 1 - Caterpillar Engine WTD vs. Alcor Predicting WTD

Reference oils are used to qualify engine test facilities and new reference oils are introduced by the Test Monitoring Center infrequently. The Alcor test has advantages over the engine test; it is cheaper ($700 vs. $15,000), quicker (1 month vs. 3 days), and more repeatable. However, the engine test provides additional information, e.g., top groove fill (TGF) and bearing weight loss. Some oils pass the WTD requirement of MIL-L-9000 G but fail the TGF limit. The MIL-L-9000 Cat 1G2 limits are WTD $\leq$350, TGF $\leq$80%, bearing weight lost $\leq$0.9 grams, and a clean piston skirt.

Pressure Differential Scanning Calorimetry

Pressure differential scanning calorimetry (PDSC) is a quick method to determine the oxidation stability of oils. A thin film of oil is heated to high temperature under pressure. The time required for an exothermic peak (due to oxidation) to appear is called the induction time and is a measure of the oil's oxidation stability. We developed an isothermal procedure which gives reasonably short test times while still differentiating among the different oils. The steps of this procedure are: (1) five microliters of sample are placed in a platinum sample pan, (2) the pressure cell is flushed with nitrogen and then brought to 185°C, (3) the cell is then flushed and pressurized with oxygen to 6.2 MPa (900 lb/in^2). Then (4) the flow is adjusted to 750 ml/min, (5) the run is started, and (6) after an exothermic peak (see Figure 2) is obtained, the data are plotted and analyzed using Dupont's Oxidative Stability V/1.0 disk program. As shown in Figure 2, the program prints out an onset time (where the thermogram starts to depart from the base line) and an induction time (intersection of the base line and the tangent to the peak inflection point).

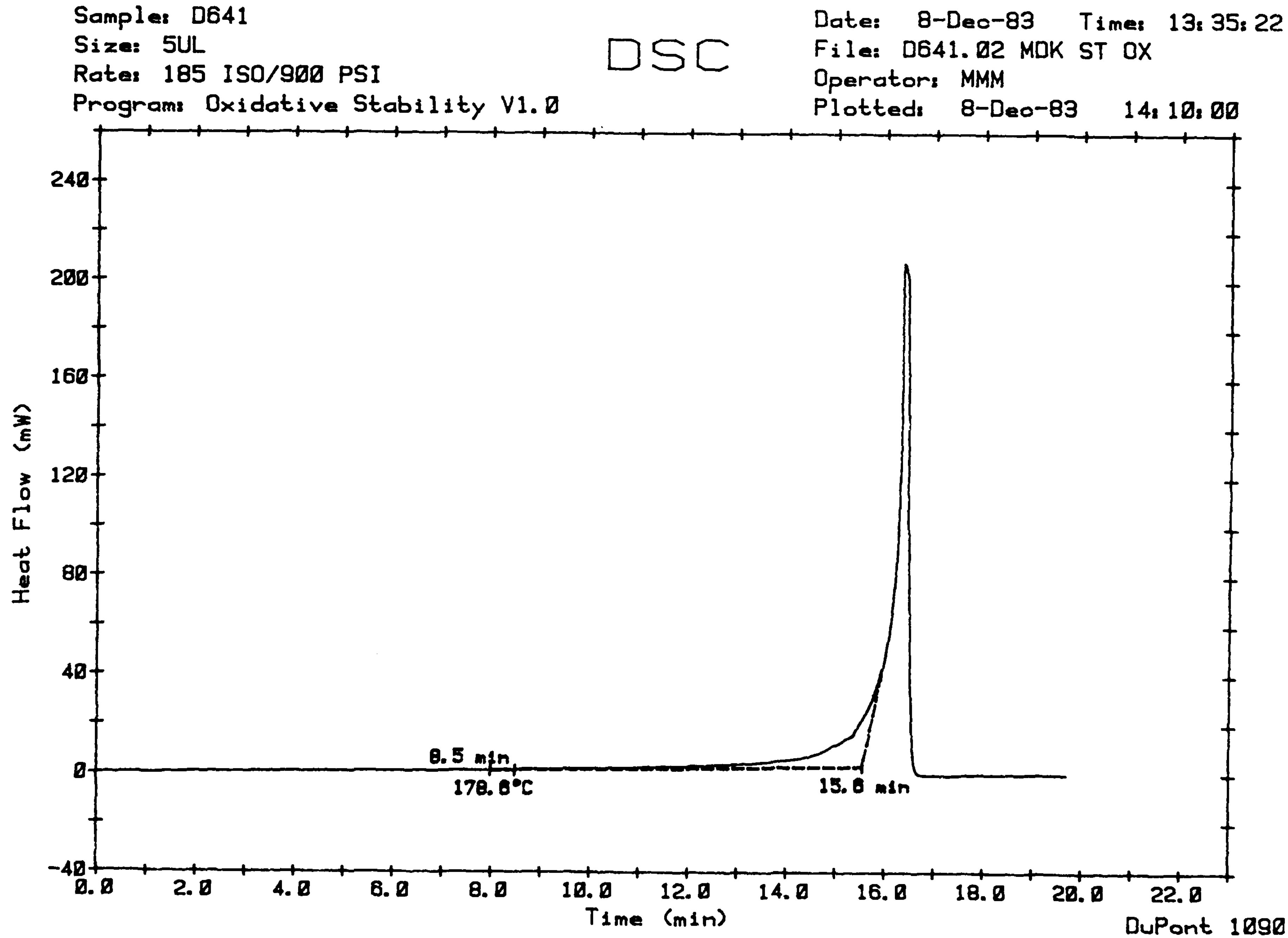

Figure 2 - Typical PDSC Curve

The induction times of available Cat 1G2 reference oils are given in Table 2 along with their WTD values. Cat engine and Alcor predicted WTD values are also plotted vs. PDSC induction times in Figure 3. PDSC induction times seem to correlate inversely with Cat 1G2 WTD values, but only five reference oils were available for evaluation. More reference oils are needed to draw any conclusions regarding the usefulness of this PDSC procedure for predicting Caterpillar 1G2 WTD values. The PDSC seems to correlate with the Alcor Deposition Test WTD but appears to have some differences with the engine test WTD for two of these oils.

TABLE 2 - PDSC INDUCTION TIMES VS. CATERPILLAR 1G2 WTD

Ref. Oil	Induction Time (minute)	Engine WTD	Alcor WTD
800	1. 49.0 2. 62.8 3. 62.7 Avg = 58.2	136.41 ±86.4*	128
801	1. 33.9 2. 33.2 Avg = 33.6	337.9 ±104.3**	260
802	1. 44.1 2. 44.9 Avg = 44.5	182.2 ±57.8**	172.6 ±8.3
803	1. 10.4 2. 15.6 3. 16.8 Avg = 14.3	394.9 ±112.4**	>400
803-1	1. 13.5 2. 13.3 Avg = 13.4	348.2 ±81.0**	390

*Weighted total demerit plus or minus standard deviation for 1G engine test.
**Weighted total demerit plus or minus standard deviation for 1G-2 engine test.

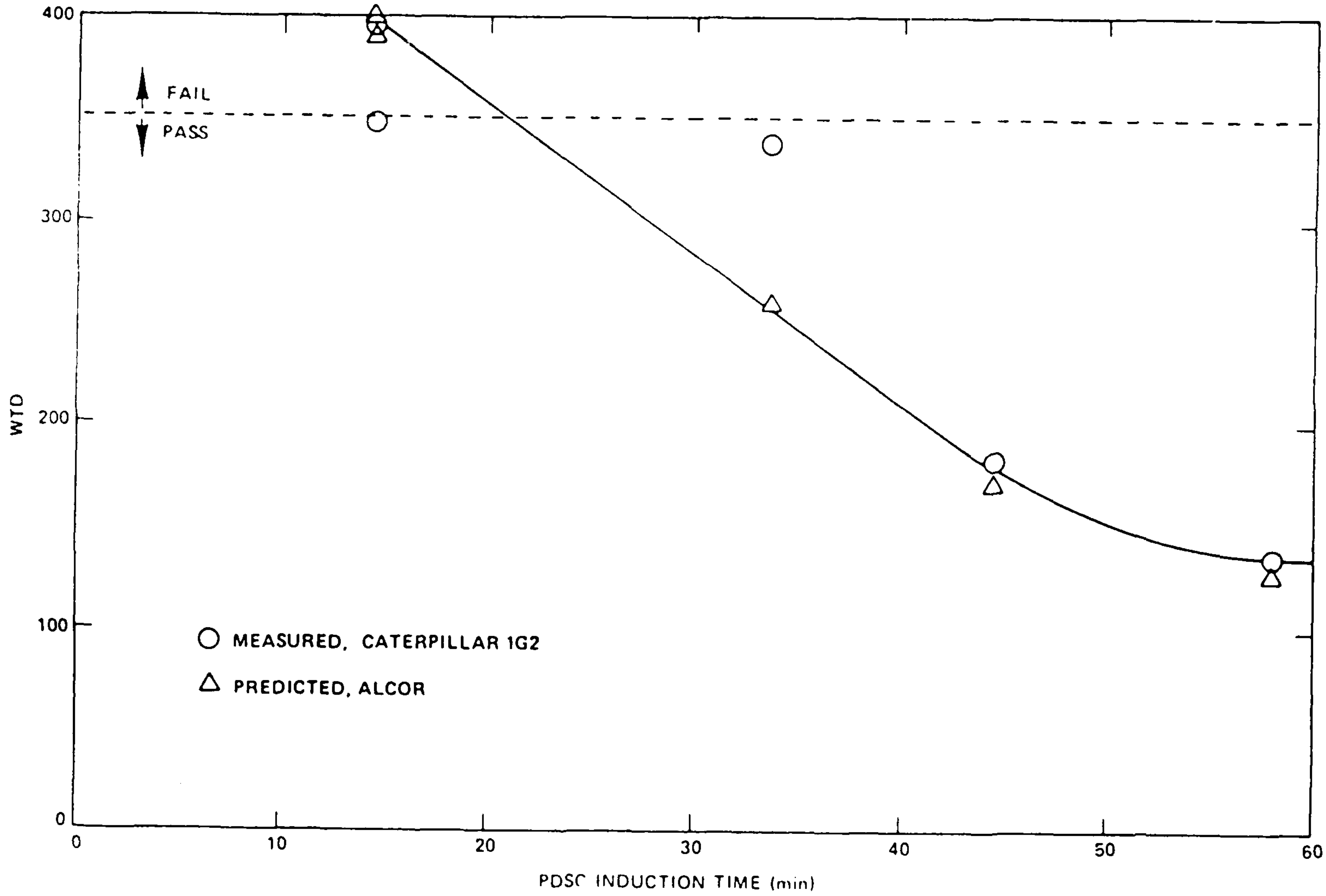

Figure 3 - PDSC Induction Times vs. WTD for Reference Oils

Microoxidation Test

The development and evaluation of a microoxidation test originated by Klaus et al[8] has been reported in detail previously. To summarize this test, a thin film of oil is placed on a cupped metal holder. The fluid holder can be made of any metal which can act as a catalyst for oxidation. A gas is blown over the oil while heating the metal holder to a temperature appropriate for the type oil being tested. The gas can be an oxidizer (oxygen or air) or inert. The former permits a measure of oxidation stability while the latter permits a measure of evaporative losses and thermal stability. After the oil is subjected to the test conditions, solvent is added to the cooled reaction tube holding the metal specimen. The resulting solution is analyzed by high performance liquid chromatography (HPLC). The analysis is compared with an analysis of the new oil to measure changes such as an increase in oxidized products and high molecular weight compounds or a loss of additives.

Normal phase, reverse phase, and gel permeation chromatography (GPC) were all evaluated as analysis techniques. GM3-71 engine samples were analyzed by a reverse phase method using: a micro-Bondapak C18 column; a linear, 2 ml/min, solvent gradient from 60/40-water/tetrahydrafuran to 100% tetrahydrofuran in 15 minutes; a 254 nanometer UV detector; and Nelson Analytical chromatography software for data analysis. The method was effective in showing increasing amounts of polar, oxidized material with increasing hours of engine operation. A few oils from the microoxidation test were analyzed by reverse phase but a GPC method gave better results. This procedure separates molecules according to size with larger molecules eluting first. The GPC method used a 100A microstyragel column and chloroform at 1 ml/min as the mobil phase.

Microoxidation conditions were based, in part, on suggested mechanisms of deposition in diesel engine oils summarized by Hsu.[9] Nitrogen dioxide (NO_2) and sulfur dioxide (SO_2) in air were used to simulate combustion gases which can form acids and mix with the oil. Also carbon black, to simulate soot from incomplete combustion, was used in some runs. The test condition most often used was 260°C for 2 hours with a 20 ml/min flow of 0.16% NO_2 + 0.16% SO_2 in-air mixture. The microoxidation test development work and results were reported previously. The two high WTD reference oils, 801(K160) and 803(D641), formed more high molecular weight materials than the low WTD reference oils, 800(K7) and 802(D640). This was based on larger UV absorption in the high molecular range from GPC analysis of the less stable reference oils.

We decided not to develop this test further since only four reference oils were available. Also the other two bench tests, Alcor 1G Deposition and PDSC, have better repeatability at this stage of development. In addition to improvement in repeatability, the micro-oxidiaton test needs further work to: (1) identify or characterize the high molecular weight materials formed and compare them with materials found in used engine oils, and (2) determine response factors and a suitable procedure for quantifying the changes in amounts of high and low molecular weight materials.

SUMMARY OF FINDINGS

Industry takes the position that engine tests are needed to predict satisfactory diesel engine oil performance even though they are costly, lengthy, and have poor repeatability. Nevertheless, improved bench tests continue to be developed and used to screen oil formulations. We have evaluated three methods that correlate with WTD values from Caterpillar 1G2 engine tests of reference oils. The Alcor Cat 1G Deposition Test predicted WTD values correlate well with the engine WTD values of seven reference oils. PDSC also gives a correlation but only five reference oils were used. The microoxidation test needs further refinement to be useful in predicting engine performance of an oil.

CONCLUSIONS

- The value of the 1G2 engine test to MIL-L-9000 is reduced by its poor repeatability and the fact that most oils which failed the 1G2 have also failed the GM3-71.

- Of the bench tests we evaluated, the Alcor 1G Deposition Test gave the best correlation with engine test results.
- Though evaluation on fewer reference oils, PDSC also appears to have value for predicting CAT 1G2 WTD values.
- The number of oils used to evaluate these bench tests has so far been too small to determine accurately their value in assessing diesel oil quality.

RECOMMENDATIONS

- If the present system of qualification at supplier expense provides enough good performance MIL-L-9000 oils, no changes are recommended for MIL-L-9000G.
- Use the Alcor 1G Deposition Test as a substitute for the Caterpillar 1G2 when (1) NAVSEA decides minor formulation changes in a qualified product do not require full requalification testing and (2) when a rapid screening test is needed for emergency procurement of a nonqualified product.
- Continue testing new 1G2 reference oils, as they become available, by the Alcor and PDSC bench tests.
- Evaluate the potential of commercial railroad and marine diesel oils and specifications for use by present and future Navy diesel engines.
- Monitor the progress by other government and industry laboratories in developing bench tests with possible Navy application.

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