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# The Development of a Thermo-Oxidation Engine Oil Simulation Test (TEOST)

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## ABSTRACT

This paper concerns a bench test developed to simulate the effect of engine operating conditions on the oxidation and deposit-forming tendencies of engine oils. The so-called Thermo-oxidation Engine Oil Simulation Test (TEOST) is carried out under temperatures and other environmental conditions identified as being significant in the internal combustion engine. These parameters can be readily modified to reflect different aspects of deposit conditions and/or different forms of the mechanical design of reciprocating engines.

The most important aspect of the TEOST is the separation of the oxidation process into the two aspects believed to be present in the engine, (1) the preparation of oxidation precursors in the so-called 'Reactor' representing the engine sump and other moderately heated areas of oil exposure, and (2) the 'Depositor' representing those areas of the engine where temperatures are such that the completion of the deposit-forming oxidation mechanism can be induced. Many variations of time, temperature, oil flow rate, cycling rate of oil temperature, and condition of oxidation precursor formation were investigated. These ultimately led to a test protocol

showing particular promise regarding the evaluation of modern engine oils.

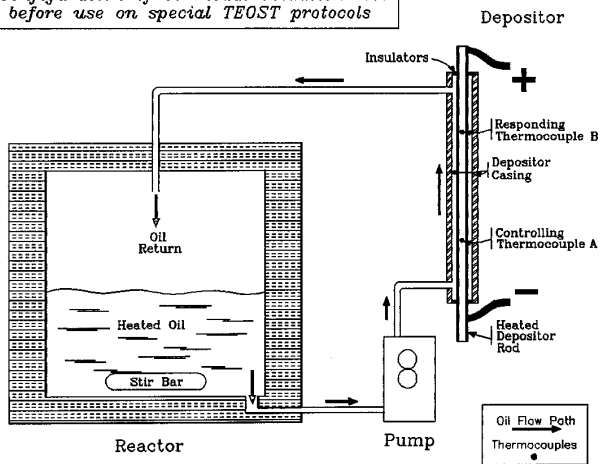
## INTRODUCTION

Deposit formation in today's modern engines is of particular concern to the engine designer/manufacturer as well as to the engine oil formulator and their mutual customer. Engine oil deposits have long been known to have a detrimental effect on the performance of the engine/vehicle in regard to drivability, durability, and, more recently, emissions.

Considerable studies by others<sup>1,2,3</sup> in the field of oxidation have shown that the primary cause of engine deposits are the relatively complex interactions that take place among the components of the blow-by gases and the engine lubricant in the presence of catalysts formed in the engine (such as particulate or soluble iron) as triggered by thermal conditions.

In order to have a meaningful test, it would seem necessary to create conditions reflecting the more important aspects of an operating engine. This was recognized in the development of the sequence engine tests over the last 35 years

Figure 1 -  
Configuration of bi-modal oxidation test  
before use on special TEOST protocols



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However, such engine tests -- as important to the automotive industry as they have been -- have brought their own problems relative to development time, costs and the rapid obsolescence of prior engine designs by newer engines demanding even higher levels of oil performance and therefore, the need for new test conditions and test beds.

## DEVELOPMENTAL EFFORTS

### First Studies

The concept of splitting depositing conditions into two regimes -- formation of the precursors at high-normal engine operating temperatures followed by a high temperature deposit-inducing zone -- was developed by one of the authors (Selby) and his associates in 1985 as a proprietary test method. In the following two years the concept was substantiated by successful application to Diesel engine lubricants designed for very high

temperature applications in the so-called 'adiabatic' engine. The setup is shown schematically in Figure 1. Thermocouple A was the temperature controlling thermocouple and Thermocouple B was the sensing thermocouple which would shut down the test whenever deposits had reached a level eroding thermostatic control of the rod temperature at which point the test automatically shut down.

### Continuing Efforts

Beginning in 1989, the authors set up an investigative strategy to move quickly through the many possible variants of an oxidation test using the number of parameters available with the prototype apparatus. Even so, based on the number of variables desired to investigate, it was obvious that developing a meaningful protocol(s) would be time-consuming at best.

On the basis of the earliest studies with the prototype apparatus on very high temperature performance oils, the parameters were systematically modified for comparative studies of engine oils regarding their performance in turbocharged engines. Such oils are not only exposed to normal engine operating temperatures but are also expected to lubricate the very hot turbocharger impeller shaft. Moreover, when the engine is turned off, the residual oil in contact with the hot shaft (especially on the exhaust side) is exposed to a static, non-replacement, condition baking at temperatures of 500°C<sup>4</sup> and more. In general, it is evident that an oil formulated to have good performance in such an environment should also be expected to perform well in other high temperature areas of the engine.

This development of an acceptable TEOST protocol and the resultant study of deposit-forming tendencies of commercial passenger car engine oils in the turbocharger was successful. The unpublished results showed the ability of the test to clearly distinguish between known good performing and relatively poor performing oils. The initial test procedure developed used a lower-temperature cycling protocol from 150° to 500°C and required about six hours to complete a test. One of the more important changes in addition to the introduction of a cycling protocol was the reversal of the controlling and sensing thermocouple locations in the Depositor tube.

Since the environment of the turbocharger also involved exposure of the oil to exhaust gases, this work led to the suggestion of one of the authors that some way should be developed to introduce such gases to the cycling oil. At the time, no readily available source was found acceptable. This changed with more recent work.

### Recent Studies

The apparent strength and versatility of the Reactor/Depositor approach to a bench oxidation test led to an effort to apply the previous investigation to the more general area of need associated with overall higher temperature engine deposits. Part of the reason for undertaking this work was related to a growing need to develop a bench test with relatively high correlation with field performance of both automotive and heavy duty engines. This was particularly important in the light of recent decisions to increase the surveillance of engine oil quality in the market place by the several technical societies associated with such concerns such as the ILSAC, SAE, API, ASTM, CMA, etc. If successful, such a bench test could

make the anti-oxidant, deposit-forming and dispersant aspects of additive and lubricant screening, formulation, and market compliance much less burdensome, expensive, and time-consuming. Moreover, this Reactor/Depositor technique with its several variable parameters also permits relatively straightforward and simple adjustment of the severity level applied to correspond with future field performance levels desired.

This paper is an introduction to, and presentation of recent work with, the technique and an outline of the direction of present and future efforts.

## APPARATUS AND TECHNIQUE

### General Comments

As shown in **Figure 1**, the basic apparatus is fairly simple. The TEOST method incorporates a sump (Reactor) that simulates the engine crankcase and contains a specified amount of the oil to be tested. The Reactor is controlled at a specified temperature to simulate the chemical conditions under which deposit precursors form. This oil is slowly pumped through the Depositor chamber.

The Depositor chamber is made up of a hollow rod or tube surrounded by a casing and the rod is heated inductively according to a program set into a temperature controller. Thus, to some degree, the Depositor can be made to simulate some chosen high temperature area of the engine which is susceptible to deposit formation. By programming the temperature controller, the rod can be cycled through a chosen peak temperature for a chosen period of time and back to the sump temperature. Circulating oil flows upward through the casing/rod annulus and thus contacts the outer surface of the heated rod directly or, as time progresses, through the

growing deposit. As may be expected, when the lubricant flows through the casing/rod annulus of the Depositor, the temperature profile experienced applies increasingly high chemical reaction stresses to the oil and additives and reaches a maximum stress about 2/3rds of the way up the annulus. This progressive reactive stress encourages these components and any precursors formed in the Reactor sump to form whatever amount of deposits are possible given the particular oxidation resistance of the formulated oil under test.

Oils will leave unique 'thermogram' on the rod related to the various reactive stages it goes through as it passes over progressively hotter sections of the rod. This 'thermogram' is rather closely related to both the chemistry and composition of the formulation.

The deposits that are formed on the rod are weighed to within 0.1 mg. Similarly, the deposits that are formed but do not adhere to the rod surface or come loose later in the test and which are carried away by the moving oil, are collected and weighed to within 0.1 mg on stacked filter disks using diluent-washed, pre-weighed filter paper. The final results are reported as the combined weight of deposits on the rod and filter.

There are two aspects which place importance on knowing the weight of the non-adhering deposits caught by the filters in comparison to the rod deposits.

1. Any deposits formed, whether attached to a surface or loose are important as a measure of the oils degree of resistance to oxidation and/or thermal decomposition.
2. Deposits which are not permanently affixed to some heated surface avoids the long term effects of such deposits in generating further growth

by obstruction of lubricant flow (such as pistons) and insulation of surfaces from the benefits of oil cooling (such as on piston under-crowns).

In comparison with other methods, the TEOST technique is unique in treating the oil simultaneously in two different but interrelated stages of 1) a location for pre-deposit precursor formation, and 2) a location where conditions are conducive to forming types of deposit associated with automotive engines.

### Specific Apparatus and Technique

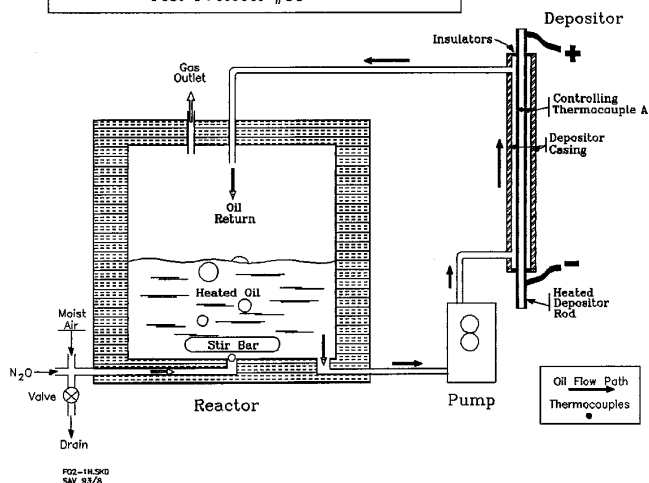
The protocol on which the data of this paper is based is called TEOST 33.

**Apparatus** - Physical layout of the unit in **Figure 2** essentially includes a sump (Reactor) which holds the test oil, a gear pump, and the high temperature zone (Depositor). Oil in the Reactor is continuously stirred. Temperature control by the prototype console is effected through use of a Glas-col heating wrap monitored by a thermocouple. Various components of interest such as reactive gases, air, catalysts, fuels, etc. required or desired in the characterization of the oxidation resistance of engine oil can be introduced either into the Reactor or the Depositor during the experiments.

Depositor rods were made of 1018 steel with fairly finely finished surfaces. The choice of steel rods was meant to reflect the various iron surfaces in the engine.

Pump speed of the prototype is variable in increments of 0.01 mL/min up to 12 mL/min. It was necessary to determine the volume of oil to effect a total change of the fluid in the Depositor, 0.8 mL, flow rates above this level in the time of one cycle were necessary to avoid

Figure 2 -  
Configuration of TEOST layout for  
Test Protocol #33



some fluid from being exposed twice to temperature spikes during one passage through the Depositor before being recycled to the Reactor. This was not thought to be conducive to a repeatable test protocol nor to give appropriate comparison of the performance of different additive chemistries. Since lower pump speeds were found to be unreliable with the motor and speed control chosen for the prototype, the minimum speed used for investigations in TEOST 33 was 0.45 mL/min. This meant an exchange five times greater than required -- a good margin of safety in Depositor fluid exchange and yet a reasonably slow circulation rate. The total volume passing through the Depositor during one full 2-hour test was about 54 mL or about half of the total volume in the system. With the stirring effected in the sump, redilution of the uncirculated oil with the incoming oil that has been exposed to the depositor conditions was believed to enhance precursor formation. Later tests confirmed this effect.

As previously mentioned, the primary source of heat to the Depositor rod

and the oil within the casing/rod annulus, was the low-voltage, high-amperage resistive heating applied to the ends of the steel rods as shown schematically in **Figure 2**. The amount of amperage supplied could be modified by the controller in response to the temperature information obtained through Thermocouple A and this relationship was used to program the temperature variation desired in the Depositor. Electrical resistivity of steel generates heat very rapidly in the rods used in these studies, on both the inner and outer surfaces of the rod. The greatest effects (deposits) of heat and temperature are near the upper third of the rod as a consequence of heat exchange with the incoming relatively cooler oil and the concentration of inductive heating effects. Therefore, the oil rising through the Depositor annulus will see a spectrum of temperatures ranging from entry temperature of somewhat less than 100°C to at least 480°C using the TEOST 33 protocol as the oil passes through heat exchange dynamics with the rod. Different additive chemistries, when observed in this procedure, show different gradations of deposits on the rod.

**Technique** - The specific technique for TEOST 33 is given in **Appendix 1**. However, it is of value to briefly discuss the most important parameters and these are noted in **Table 1**.

**Temperature Profile** - The temperature range and cyclic program is shown in **Figure 3**. It covers a range of 200° to 480°C after the first cycle is initiated from ambient temperature. The authors believed that cyclic conditions would simulate more closely the variable temperatures encountered by hot surfaces in the engine, particularly the relatively brief temperature spikes that occur

Table I

TEOST 33 PARAMETERS

- Rod temperature controlling thermocouple - TC# 5 (Tc/T2)
- Pump speed - 39.5 sec/revolution, approximately 0.45 mL/minute of oil flow through the depositor (monitored every 10 minutes for accuracy).
- Rate of air flow - 3.6 mL/min. through H<sub>2</sub>O.
- Rate of N<sub>2</sub>O flow - 3.6 mL/min. through H<sub>2</sub>O.
- Reactor temperature - 100°C
- Reactor oil volume - 100mL. There is an additional 15mLs of oil in the lines. At this point add Iron Napthenate to make up 100 PPM in the oil.
- The pump is left on continuously to eliminate warmup effects.

TEOST TEST CYCLE

<u>Program Step</u>	<u>Temperature</u>	<u>Time</u>
0	Ramp to 200°C	Immediate
1	Hold at 200°C	1 minute 15 seconds
2	Ramp to 480°C	1 minute
3	Hold at 480°C	2 minutes
4	Ramp to 200°C	4 minutes
5	Hold at 200°C	1 minute 15 seconds

Cycle 1 goes through steps 0 - 5 once, cycles 2 - 12 go through steps 1 - 5, 11 times. Each cycle takes 9.5 minutes and the total test time is 114 minutes.

which are followed by the washing of the surface by oil under somewhat cooler conditions.

**Number of Cycles** - The test is completed with 12 cycles of approximately 10 minutes per cycle or two hours per test. This number of cycles and the overall test time is one of the most readily available variables to fine tune the test. For the work of TEOST 33, a 12-cycle test protocol produced interesting and correlative data when coupled with the above temperature profile.

**Sample Volume** - The total oil volume required for one TEOST test is 250 mL. The actual sample volume to be tested includes 100 mL oil in the

reactor plus 15 mL left in the lines after thorough flushing with the new test oil. This volume was chosen to give sufficient residual volume after test for other relevant analyses while minimizing the amount of initial test oil to leave sufficient fresh oil for other tests as well. Another consideration was to have a volume sufficiently large that the recirculated oil from the Depositor would have some, but not overwhelming, influence on the chemistry occurring in the Reactor.

**Sample Flow Rate** - Test oil is passed through the Depositor annulus **Figure 4** at a flow rate of 0.45 mL/minute. Essentially, with the volume of the filled Depositor (~0.8 mL), the flow



FIGURE 3 -

TEOST 33 Temperature Profile

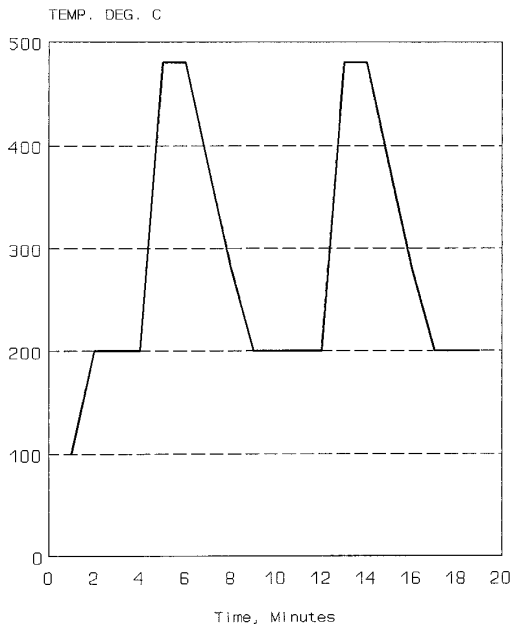
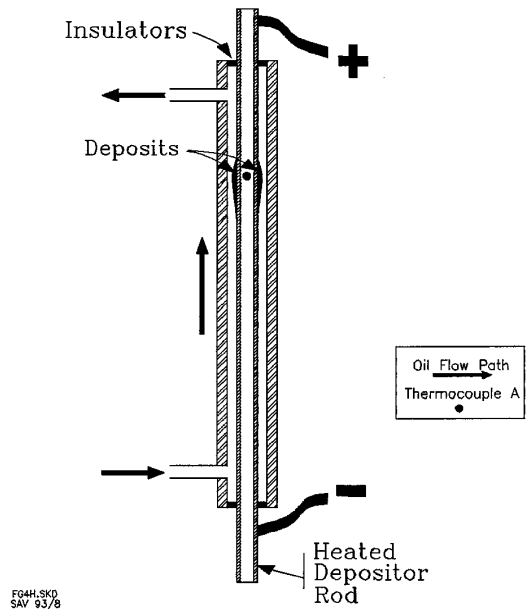


Figure 4 -

Sketch of Depositor showing locations of thermocouple and deposits



rate was considerably higher than that needed to remove the oil exposed to the temperatures of the previous cycle. Thus, on each new cycle, the oil chemistry involved in deposit formation reflected the condition of oil from the Reactor.

**Rate of Moist Air Flow** - Moist air is supplied in excess of any reaction rate at approximately 3.6mL/min. The author's recognized fairly early in the test protocol development that simulation of the engine operating conditions should involve moist air -- moisture from the products of combustion and air as a source of oxygen to replace that depleted by the oxidation reactions.

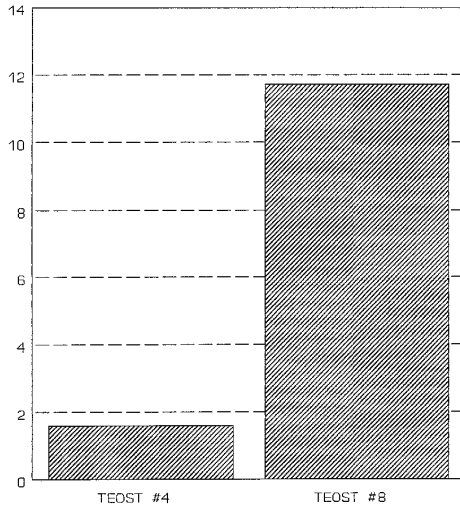
**N<sub>2</sub>O Gas Flow** - Nitrous oxide, N<sub>2</sub>O, is supplied in excess of any reaction rate at the same rate as moist air. In lieu of the availability of stronger and more

precaution-demanding oxides of nitrogen such as NO and NO<sub>2</sub>, the author's used the readily available and safe N<sub>2</sub>O. However, reactivity of this gas becomes significant only above 300°C and although used in TEOST 33, was felt to be of more limited value compared to NO and NO<sub>2</sub> in providing some of the nitrogen-oxide driven reactivity desired. However, earlier work with the TEOST 8 protocol showed that there was a significant increase in severity with the addition of N<sub>2</sub>O to the TEOST protocol as seen in **Figure 5**.

**Reactor Temperature** - Reactor temperature was held at 100° ± 1°C during the entire analysis of a test oil to reflect the approximate sump temperature in the automotive engine.

FIGURE 5 -

CRO-2 Reference  
TEOST #4 vs TEOST #8



TEOST #4 = Air, no N2O  
TEOST #8 = Air with N2O

## REFERENCE OILS USED IN TEOST 33 STUDIES

In the development of bench tests on lubricants it is always desirable to correlate results with known field performance of these oils. The following four reference oils were selected based on their performance in customer usage. These Chrysler Reference Oils are shown elementally in **Table 2** and are identified as CRO-1, 2, 3, and 4.

**CRO-1** was a commercially available SAE 10W-40 SF engine oil that was associated with several turbocharger field failures in turbocharged engines.

**CRO-2** was a commercially available SAE 10W-30 SF/CD engine oil with proven good performance in naturally aspirated and turbocharged engines.

**CRO-3** was a reference oil used in Europe and identified as a

'High' performance reference oil in proprietary turbocharged vehicle tests.

**CRO-4** was a reference oil used in Europe and identified as a 'Low' performance reference oil in proprietary turbocharged vehicle tests.

## RESULTS

One of the first comparisons of data using the TEOST 33 protocol was encouraging. This data is shown in **Figure 6**. Total rod and filter deposits are given. It is evident that a strong distinction is made between good and poor engine oils. The range of performance is from 17 mg for CRO-3 (a high-performing oil) to 77.2 mg for CRO-1 (a field-failing oil). All four CRO oils are ranked consistently in regard to engine/field performance.

### Addition of Iron Catalyst

Available literature <sup>5</sup> on the subject of oil oxidation suggests that soluble iron can act as a catalyst to enhance oxidation of oil. Additionally, this literature suggest that oil containing copper as an oil additive may not perform well in the absence of soluble iron. **Figure 7** shows the performance of several reference oils including oils identified as EXP-1 and EXP-2. These oils contain the same performance package except that EXP-2 contains the copper additive while EXP-1 does not. These oils were treated by adding a soluble iron compound (iron naphthenate) at a level of ~100 PPM elemental iron to one of two samples of each oil. The results tend to substantiate the literature particularly when it is recognized that oil oxidation precedes deposit formation. Reference oils EXP 2 and CRO-1

Table 2

## Chrysler Reference Oils

### Elemental Composition

Element	CRO-1	CRO-2	CRO-3	CRO-4
Sulfur % wt.	0.36	0.87	0.42	0.11
Nitrogen % wt.	0.04	0.10	0.11	0.07
Boron, ppm	100	----	160	----
Zinc, ppm	1200	1040	1240	920
Phosphorus, ppm	1100	960	1070	910
Magnesium, ppm	1400	400	----	----
Calcium, ppm	----	980	2200	1730
Copper, ppm	100	----	----	----
Sodium, ppm	----	750	----	----

contain different levels of a copper anti-oxidant and the literature<sup>5</sup> points out that the response of the copper additive is concentration dependent. It would appear that copper at a level of approximately 90 PPM (CRO-1) is unaffected by the addition of iron. However, the EXP 2 oil contains approximately 190 PPM and directionally appears to improve with the iron addition. Oil CRO-4, the low-performance oil, has no copper additive but may have reacted more severely because of its lower additive treatment level in combination with the iron acting as an oxidation catalyst.

#### TEOST 33 Repeatability Study

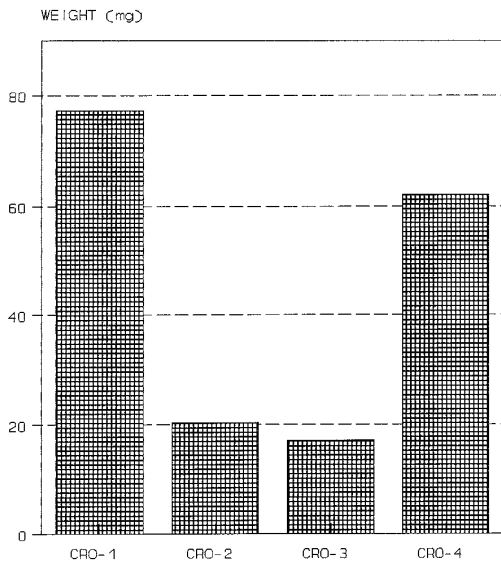
Repeatability is one of the first concerns in the development of a bench test. **Figure 8** demonstrates

the repeatability of TEOST 33 using Reference Oil CRO-2. The statistical mean value for this set of 17 analyses is 20.1 mg and the Standard Deviation is 1.16 mg. For an oxidation test requiring the measurement of deposits to tenths of a milligram, this seems to be an acceptable level of precision and, to some extent, reflects the effort which went into development of the protocol used.

#### DISCUSSION

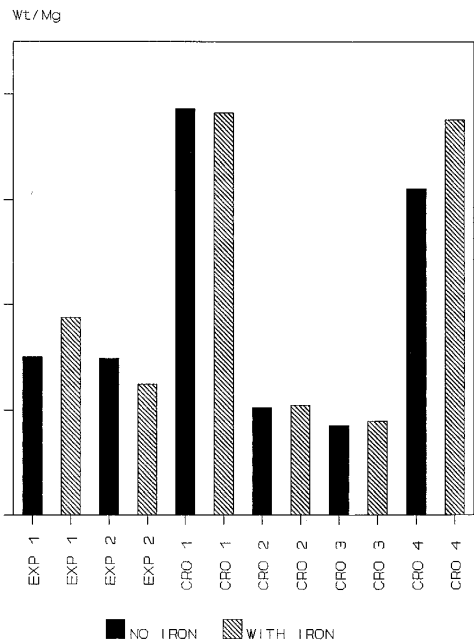
The reference oils used for development of the TEOST Protocol 33 were chosen because of their relatively good and poor performances in the high temperature areas of the turbocharger on engines so equipped. In comparison, the Sequence III results on the CRO-1 and CRO-2 reference oils showed similar passing characteristics and

FIGURE 6 -  
TEOST 33 Reference Oil



TOTAL DEPOSITS

FIGURE 7 -  
TEOST 33 - Total Deposits



did not differentiate between the two oils relative to piston deposits.

The TEOST procedure is being proposed to define future engine oils for use in engines that will most likely be operating at considerably higher temperatures, particularly in the piston/bore areas. Such performance, in addition to meeting future emission standards requiring certification of vehicles as meeting emissions standards for 100,000 miles, makes the need for more deposit resistant engine oils critical as well as the need for suitably rigorous and correlative bench tests.

## SUMMARY

This paper has presented the developmental background of an

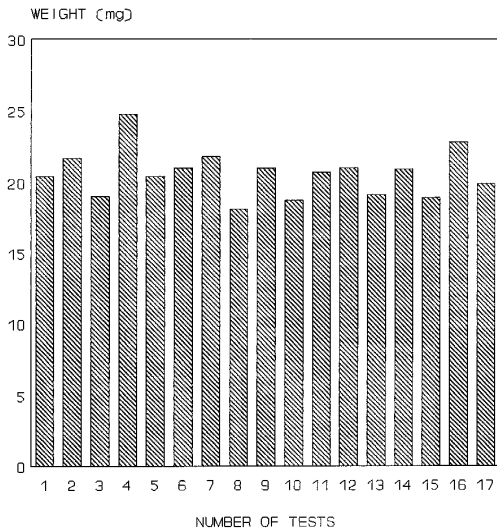
accelerated bench test that simulates the environment of a fired engine. Preliminary studies simulating high temperature conditions indicate that the method is capable of discriminating between good and poor performing engine oils, based on the oil's ability to resist deposit formation in critical areas of future engines.

## FUTURE WORK

Future work is aimed at developing a different temperature profile in line with upper piston operating temperatures (260° to 300°C). In view of the effect of the influence of the metal surface on the chemistry of deposit formation, it is intended to use an aluminum surface or rod of an aluminum alloy similar to that used in fabricating the piston. Similarly, considering

FIGURE 8 -

TEOST 33 - Repeatability Study  
With CRO-2 Reference



TOTAL DEPOSITS  
STANDARD DEVIATION: 1.16  
MEAN: 20.1

the influence of blow-by gases on piston deposit formation, it is planned to introduce a mixture of these gases ( $\text{SO}_2$ ,  $\text{CO}$ ,  $\text{NO}$ ) in addition to  $\text{H}_2\text{O}$  and ( $\text{O}_2$ ). Additionally, the authors plan to refine the prototype apparatus on the basis of the experience thus far gained.

## ACKNOWLEDGMENTS

The authors wish to express their appreciation for the hard work of several individuals who worked through the various techniques to produce a repeatable series of protocols involving fine weighing techniques and close control of the apparatus. Particularly, the authors would like to thank Jennifer Richardson and Brian Cluff of the Savant Laboratories for their dedication.

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## APPENDIX 1

### TEOST 33 SET-UP PROCEDURE

- I. It is essential that proper and consistent clean up of the test bed take place prior to each and every TEOST run.
  1. Clean the **reaction** vessel - Rinse the gas/air inlet ports and the interior of the vessel with hexane. Be sure to remove any particles which cling to corners and crevices. Dry thoroughly with a paper towel. Complete the drying process by applying a vacuum to a gas/air inlet nipple for no less than 3 minutes.
  2. Clean the stainless steel tubing (pump inlet, pump outlet and return tube). Rinse hexane through the tubes and allow to dry thoroughly while attached to a vacuum source.
  3. Wipe clean with a paper towel all old test oil from tubing connections and remove all old Viton O-rings. Clean the parts of the tubing where the O-rings were in contact.
  4. Replace all O-rings with new Viton O-rings.
  5. 250 mL of oil is measured into a beaker. Then 417.5 uL Ferric naphthenate (in mineral spirits) with 6% iron content is added via a syringe. A stir bar is added and the beaker is placed on a magnetic stir plate for approximately 15 minutes.
  
- II. Old test oil in the **pump** must be replaced by using a flush method.
  1. Connect the reaction vessel to the pump inlet tube.
  2. Fill the reaction vessel (with swing arm in the up position) with 250 mls of test oil.
  3. Place a graduated flask (enough to hold 200 mls) beneath the pump outlet tube to collect the flushed fluid.
  4. Start the pump by pressing the "pump" button on the control council. The pump speed should be set at the maximum setting (999). Let the oil flow until 150 mls has drained into the graduated flask.
  5. Stop the pump and dispose of the flask contents into the proper waste receptacle.
  
- III. The proper preparation of the **depositor rod** is essential for repeatable results.
  1. Clean the depositor rod casing by rinsing it with hexane and brushing the interior with a .22 caliber brass brush.
  2. Conduct a final rinse with hexane.
  3. Dry the depositor rod casing thoroughly by attaching to a vacuum source.
  4. Select a depositor rod from the desiccator. Be sure to pull a vacuum on the desiccator for storage of the remaining depositor rods.
  5. Rinse the depositor rod with hexane, this removes a protective coating applied at the manufacture.

6. Scrub the interior of the depositor rod with a long pipe cleaner. Be sure to soak the pipe cleaner in acetone while it is pulled through the rod. Use a second acetone soaked pipe cleaner to clean the interior of the rod of any residual material.
7. Buff the rod with very fine (0000) steel wool to remove any additional residue.
8. Conduct a final rinse of the depositor rod with acetone.
9. Dry the interior of the depositor rod thoroughly by applying a vacuum.

NOTE: Care should be taken to prevent contamination of the rod from grease and oils in human skin. Washing your hands with soap and hot water prior to handling the rod will prevent this.

10. Weigh and record the mass of the depositor rod.

#### IV. The depositor assembly

1. Insert the prepared depositor rod into the cleaned depositor rod casing.
2. Place a new viton o-ring over each end of the depositor rod protruding from the depositor rod casing.
3. Center the depositor rod into the casing by looking through the thermocouple inlets on the side and adjusting the rod until the indented radius is visible.
4. Place the "top-hat" sealers over both exposed ends of the rod and screw the nut in place finger tight.
5. Place the assembly into the proper position on the Alcor test bed being sure that some stainless steel is visible at both the top and bottom holders.
6. Tighten the hex screws with an allen wrench. Sufficiently tighten to ensure a good contact surface.

#### V. Complete the flow circuit.

1. Insert the stir bar into the reaction vessel and check to ensure that the bar is spinning. If not, turn the test bed (Alcor) power on and off in succession until the bar spins.
2. Hook up the return tubing by making the proper connections and tightening with a 7/16 inch wrench.
3. Connect the pump outlet tube to the depositor assembly finger tight.

#### VI. The thermocouples now need to be set.

1. Screw thermocouple B into the top end of the assembly finger tight.
2. Screw in thermocouple T1 into the bottom end of the assembly finger tight.
3. Set thermocouple TC/T2 (controlling thermocouple) at the at 50 mm into the center from the top of the depositor rod.
4. Insert thermocouple T2/TC into the center from the bottom of the depositor rod until it meets TC/T2.
5. Reset thermocouple TC/T2 at 22 mm.

VII. Make final preparations before starting run.

1. Tighten all thermocouple and tubing fittings.
2. Make sure that 100 mls of test oil are in the reactor vessel.
3. Place the heated jacket over the reaction vessel and fasten with velcro. Push the red heater button on the tower behind the pump. This will begin to preheat the test oil to 100°C.
4. Recheck the flow circuit to make sure everything is connected properly.
3. Connect the N<sub>2</sub>O and air hoses to the nipples of the swing arm of the reaction vessel.
4. Open the N<sub>2</sub>O tank valve and the t-valve of the air supply. Both should be set at 1 bubble per second.
5. Start the pump at the 999 setting until oil flow is established at the top of the reaction vessel. Once flow is established set the pump speed down to 59.
6. Set the thermocouple switch on the side of the tower to "D", this reads the reaction vessel temperature and displays it on the LCD readout on the control module.
7. Wait for the LCD to read 100°C.
8. Be sure the ink pens are down on the recorder and that it is set to 6 cm/hr chart speed.
9. Measure the time it takes for the pump gear to make one complete revolution. Adjust the pump speed control to get approximately 39.5 seconds per revolution.
10. Go into the tune mode on the test bed controller and check the PID settings and the program to make sure they are correct. (PID settings: PR Band = 80%, Reset = 4.50, Rate = 0.01)
11. Conduct one final check of the system for any misconnections or potential problems.
12. Turn on the strip chart recorder to 6 cm/hr.
13. Push the heat button on the control module.
14. Push the start/stop button on the temperature controller.
15. Record the start time on the data sheet.
16. Set a timer for ten minutes. After 10 minutes recheck the pump by timing one and then two revolutions using split timing for accuracy. If the pump is not maintaining close to 39.5 sec/revolution then adjust the pump speed. Pump speed is critical and should be checked every ten minutes.



## TEOST - BREAKDOWN

### I. Test completion

1. Shut down the (Alcor) test bed.
2. Hit the two heat buttons, one the tower and one on the control module to turn off the heat source.
3. Hit the main power switch on the control module.
4. Remove the air and N<sub>2</sub>O gas lines from the swing arm nipple and turn the supply off.
5. Move the chart speed on the recorder to "off" and lift up the pens.

### II. Collect the test oil.

1. Remove the heating jacket from the reaction vessel
2. Move the reaction vessel's swing arm down to drain the test oil into a 500 ml flask. Allow oil to drain out.
3. Detach the pump inlet tube from the reaction vessel and cap the opening to the reaction vessel.
4. Rinse the reaction vessel thoroughly with hexane and allow to drain into the 500 ml flask. After draining, return the swing to its upright position.
5. Remove the thermocouples from the depositor casing being sure to catch the drops of oil, that will initially come out, into the flask.
6. Remove the pump outlet tube from the depositor casing. Again, be sure to catch any oil into the 500 ml flask.
7. Detach the return tube from the depositor casing and the cover from the reaction vessel.
8. Rinse the return tube with hexane into the 500 ml flask.
9. Rinse the pump outlet and inlet tubes with hexane into the 500 ml flask.
10. Loosen the hex nuts on the bus bars and remove the depositor casing.
11. Hold the depositor rod casing (containing the depositor rod) over the 500 ml flask and rinse with hexane through each opening.
12. Loosen the nuts (2) on the casing and remove them along with the "top hat" sealers.
13. Use a dental pick tool to gently remove the o-rings that are embedded between the depositor rod and the depositor casing.
14. Gently pull the rod out of the depositor casing while holding it over the 500 ml flask to catch any deposits that may have been loosened during the disassembly.
15. Rinse the depositor rod over the 500 ml flask carefully with hexane. It is important to remove all the oil that is in the crevices of the deposits. Any remnants of the test oil will add weight to the rod. (now is a good time to weigh the depositor rod - see section three)
16. Rinse the interior of the depositor casing into the 500 ml flask.
17. Rinse the top of the reactor into the 500 ml flask.

III. **Weigh** the depositor rod

1. Use vacuum to thoroughly dry the depositor rod using the same technique as listed in the set-up section.
2. Weigh and record the mass of the depositor rod.

IV. **Filter** the test oil (refer to appendix A before proceeding)

**NOTE:** It is sometimes necessary to add hexane to the mixture in the 500 ml flask to decrease the viscosity of the fluid. It is easier to filter and less likely for oil to be trapped in the filters.

1. Place five 10-micron filters in a plastic filter holder with the cover on.
2. Weigh and record the mass of the holder and filters.
3. Clean the components of the vacuum and filtration device.
4. Assemble the vacuum filtration device. Make sure that dry ice is surrounding the cold trap. Hexane vapors will quickly ruin a vacuum.
5. Place all but one of the pre-weighed filters in the vacuum filtration device. Use a clamp to hold the top in place.
6. Start the vacuum.
7. Rinse the filters with hexane, then pour the contents of the 500 ml flask slowly over the filters.
8. Rinse out the flask and the sides of the filtration device with hexane.
9. Carefully use your hand to cover the filtration device while squirting hexane through the seal where the filters lie. This will help move the deposit particles toward the center of the filter paper. Be careful to remove your hand slowly (thus removing the vacuum slowly) or deposits will "jump" to the sides and force you to repeat step #8.
10. Run a good amount of hexane through the filters to clear them of any residual test oil. Again, any oil remaining in the filters will give higher weights and distort the results.
11. Place a loose cover over the top while the vacuum is running. Leave this in place for 1/2 hour (using a timer).
12. At the end of the 1/2 hour, carefully disassemble the vacuum filtration device.
13. Carefully place the last filter on top of the deposits on the other to hold the particles in place while the filters are carefully slid into the filter holder.
14. Crack the cover a bit and place inside the vacuum desiccator (be sure the desiccating chamber does not contain any desiccant).
15. Connect the vacuum desiccator to the vacuum.
16. Turn on the vacuum and allow the system to dry the filters for ten minutes (use the timer)
17. After the ten minutes is up, slowly replace the atmosphere in the desiccator and remove the cover.
18. Firmly close the cover to the filter holder.

V. **Weigh** the filters

1. Carry the filters to the balance and record the mass.

VI. **Final** results

1. Calculate the weight of the deposits on the rod and on the filters and add them together for the result of the test. The results are recorded in 0.0 mg.
2. Label the depositor rod's casing and the filter holder as TEOST test # and oil identification code.
3. Place reduced copies of the strip chart and data sheet into a test log with any comments on the run.
4. Use the roto-vap to strip the hexane off the filtrate. Keep the test oil and store in a plastic bottle for future reference. Label the retained test oil the same as in step #2.

**APPENDIX A TO PROCEDURE**

The ten-micron filters should be pretreated before filtering the test fluid. The pretreatment should be conducted in the following manner.

1. Place five 10-micron filters into a plastic filter holder.
2. Record the initial weight on the data sheet.
3. Assemble the vacuum drying device and place the filters into it.
4. Use hexane to "clean" the filters in order to remove any factory place hexane solubles.
5. Place the "cleaned" filters into the metal spiral holder in the desiccant chamber and apply a vacuum for 60 minutes.
6. Take the final weight and record.
7. These filters are now ready to collect the deposits per the TEOST procedure.

**INSTRUMENTAL CONSIDERATIONS**

Reaction Gases: The air and N<sub>2</sub>O are bubbled through a container filled with water. The rate of each should be 3.6 mL/min.

**ADDITIONAL SETTINGS**

1. Pump speed setting is set at 39.5 sec/rev or 0.45 ml/min.
2. Reaction vessel temperature is controlled at 100°C.
3. Oil volume in the reactor is 100 ml (there is an additional 15 mls of fluid in the lines)
4. Rate of air flow is 3.6 mL/min.
5. Rate of N<sub>2</sub>O flow is 3.6 mL/min.

- 6. PID settings: PR Band @ 80%  
Reset @ 4.5  
Rate @ 0.01
- 7. Power settings: Bias 10% 45°C 6 VAC  
Span 90% 520°C 40 VAC

**TEMPERATURE PROGRAMMING**

The temperature program on the LFE controller for TEOST testing should read as follows:

SET POINT 0	200
TIME 1	1:15
SET POINT 1	200
TIME 2	1:00
SET POINT 2	480
TIME 3	2:00
SET POINT 3	480
TIME 4	4:00
SET POINT 4	200
TIME 5	1:15
SET POINT	200
TIME 6	0.00

# OF CYCLES 12