A Laboratory Method for Measuring Bulk Volatility of Engine Oils - Comparative Results

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ABSTRACT

Previous studies to improve upon the Noack volatility test have reported a new approach which does not require toxic Wood's Metal for heating yet agrees well with Noack test results. In addition, the new approach collects 99% of the volatilized oil for optional analysis. This can be important apropos to phosphorus levels which are of concern regarding automotive exhaust catalyst life. To more closely compare the new approach with the Noack test, reference oils used in a recent ASTM volatility round-robin study were analyzed and the new approach was found to produce close agreement with the Noack technique and generally greater repeatability.

KEY WORDS

Volatility, bulk volatility test, Noack test, engine oil

GLOSSARY

Oil Consumption - Any loss of oil volume in an engine by either leakage, evaporation, or by means of chemical alteration (i.e. deposits, varnish, sludge).

Oil Evaporation - Internal oil loss by heat-facilitated volatilization of engine oil.

Oil Leakage - External oil loss by leakage past gaskets and seals.

Bulk Volatility - The measured volatility of a bulk quantity of unagitated oil in contrast to gas chromatograph volatility measurement from a relatively small number of agitated molecules.

R^2 - Coefficient of Determination - a measure of interdependence of two variables where a value of unity indicates complete interdependence and values less than unity indicate the degree of interdependence from zero to unity.

INTRODUCTION

EFFECTS ON ENGINE OILS - Temperature is a major factor in facilitating the evaporative loss of some lower molecular weight components present in fully formulated engine oils. Although oil can experience very high temperatures (for example, valve heads can reach 750°C[1]), normal oil service temperatures in the summer may range from ambient to 300° C[2].

It has been reported that up to one-half of total oil consumption may occur by evaporation in the valve assembly[3]. Such evaporative loss will increase both 1) oil viscosity, and 2) the rate of oil consumption. Although these effects are generally considered negative, there are some positive aspects.

The increase in the oil's viscosity increases bearing oil's film thickness. Also, oil consumption often leads to "topping off" the oil with fresh lubricant thus somewhat recharging the additive content of the oil in the engine -- a view which has led to comments that a lack of oil consumption may cause the engine to wear rapidly due to the oil having greater susceptibility to chemical changes[4].

PHOSPHORUS EFFECTS - Questions, however, are being raised within the automotive manufacturing industry and elsewhere regarding the possible effect engine oil volatilization has on the catalyst present in the catalytic converter. In particular, phosphorus has come under scrutiny for its possible role as a component of the engine oil volatiles[5,6,7]. Since the catalytic converter helps reduce the amount of pollutants that reach the atmosphere, the need to understand more about that portion of the oil components which are lost to volatilization may be of considerable importance regarding catalyst life and possible effects on the atmosphere.

NOACK EVAPORATION TEST - Dr. K. Noack first published his work in 1936[8]. Since then, the Noack volatility test has become an industry standard for determining relative volatilization rates for engine oils[9,10]. This test requires evenly heating an unagitated mass of oil by immersing the evaporation chamber in molten Wood's Metal (see Figure 1).
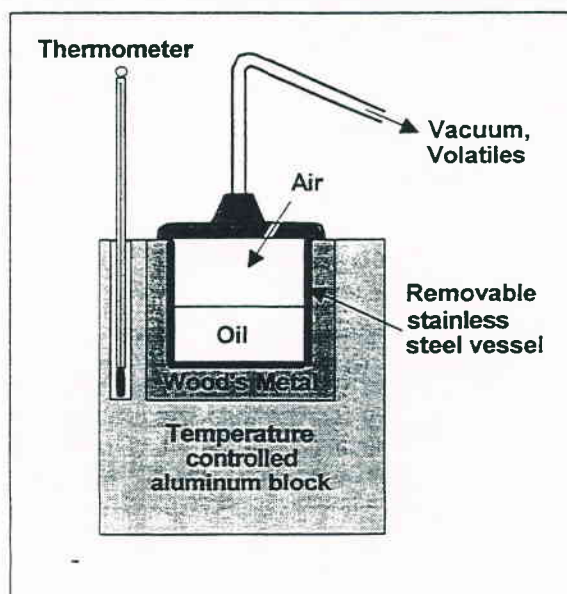

Figure 1: Basic diagram of original Noack volatility test showing Wood's Metal bath

Volatiles are removed from the heated vessel with air flow induced by a small amount of vacuum. Although molten Wood's Metal acts as an excellent heat transfer medium, it is a concern because of the hazard of possible toxic fume production while in the molten state[11,12]. Burns from handling the hot molten Wood's Metal is another concern, especially when it is necessary that the Wood's Metal bath be full and overflowing to minimize repeatability errors.

The foregoing safety concerns at the authors' laboratory prevented setting up the Noack test apparatus with its requirement for the use of Wood's Metal. However, it was appreciated that the Noack test, being a bulk volatility test (in contrast to simulated distillation by gas chromatography) produced information not obtainable by the latter test. Therefore, it was desirable to modify the Noack method by removing the need for use of Wood's Metal as a heat transfer medium. Redesign of the test equipment also gave opportunity to modify the evaporation chamber in an effort to improve repeatability. More important, this redesign provided an opportunity to develop a collection technique for the volatiles otherwise lost in the Noack method. Collection of volatiles was considered a major benefit in studying the pattern of phosphorus volatility and other matters related to the volatiles.

NEW METHOD - Previous papers have introduced the initial prototype instrument of this new approach[13,14] which retained the main volatilization parameters of the original Noack volatility test: that is, 250° C test temperature, one hour test time, uniform heating surface, and a similar surface area.

Wood's Metal was eliminated from the method by using a special noble metal sheathing bonded to the volatility cham-
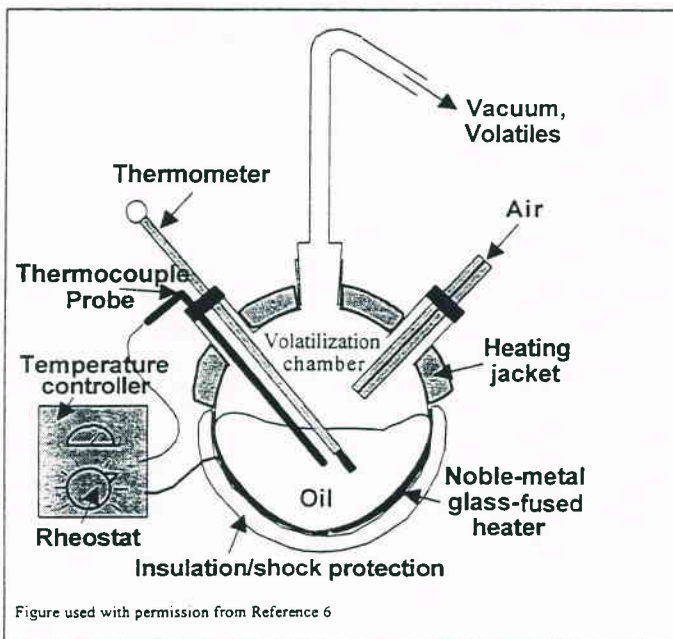

Figure 2 - Sketch of original prototype apparatus

ber as shown in Figure 2.

The temperature of the test fluid was controlled with a thermocouple directly immersed in the oil. Flow rate of air through the volatility chamber during the test was controlled to 2.8 Liters/minute by adjusting the amount of vacuum in the volatilization chamber.

Correlation between the original Noack test and this prototype apparatus was reported to be good with $R^2=0.991$, a slope of 0.93 (1.00 is perfect), and an intercept of 0.09% (zero is perfect). However these were preliminary results and

lacked rigorous repeatability data. Moreover, the method used controlled air flow rather than controlled vacuum. Consequently, it was thought that improvements could be made particularly in collecting more of the volatilized oil. The areas of temperature control, air introduction, vacuum control, volatile collection, and method simplicity were all examined during this further research.

For descriptive purposes, the new method this paper is presenting will be referred to as the 'bulk volatility test' while the prototype presented in the previous paper will continue to be called 'prototype', and the original Wood's-Metal-dependent method simply 'Noack test' or 'Noack volatility test'.

EQUIPMENT

BULK VOLATILITY TEST - Research done on the new method since the previous publications[13,14] has given considerable information regarding improvement of the sensitivity and precision. For example, design changes were made in the technique of air introduction, precision of temperature control, and the process of coalescing and collecting volatiles.

Air Introduction - As in the original Noack method, a slight vacuum (20 mm H_2O) generates a flow of air through the evaporation chamber or 'volatility chamber'. The air volume for these tests was controlled through a 0.063 inch di-
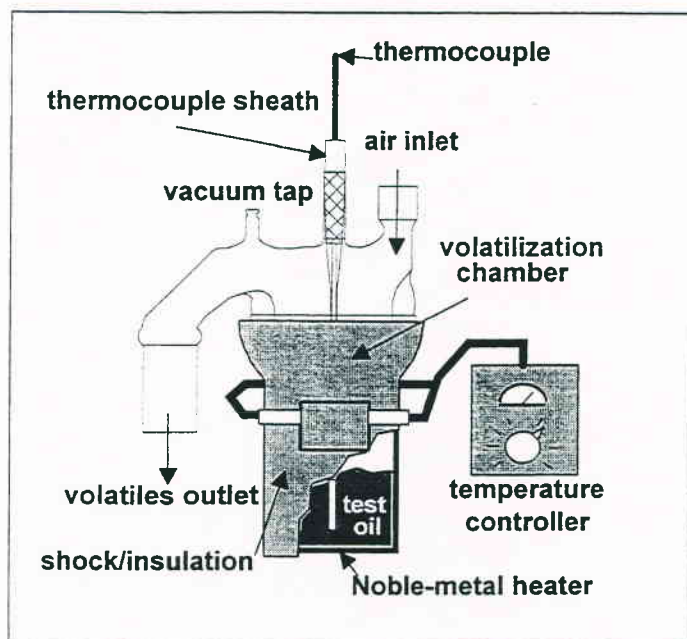

Figure 3: Volatilization chamber set-up showing thermocouple sheath, vacuum tap, and air inlet/volatiles outlet

ameter orifice tube 0.53 inches in length.

Temperature Control - As noted earlier in the paper, the oil inside the bulk volatility test's volatilization chamber is controlled to 250 ± 1° C for one hour. Experience with the prototype unit had shown that repeatable thermocouple positioning was very important for repeatability. To accomplish this, a stainless steel sheath was designed to be inserted into a receiver on the reaction flask top. The sheath directs a custom thermocouple to exactly the same position in the oil for every test as shown in Figure 3. This J-type thermocouple is only one-sixteenth of an inch in diameter and has a grounded junction to facilitate a relatively quick response time.

Careful sealing of all joints assured that the flow of the vacuum-induced air was only through the air-introduction
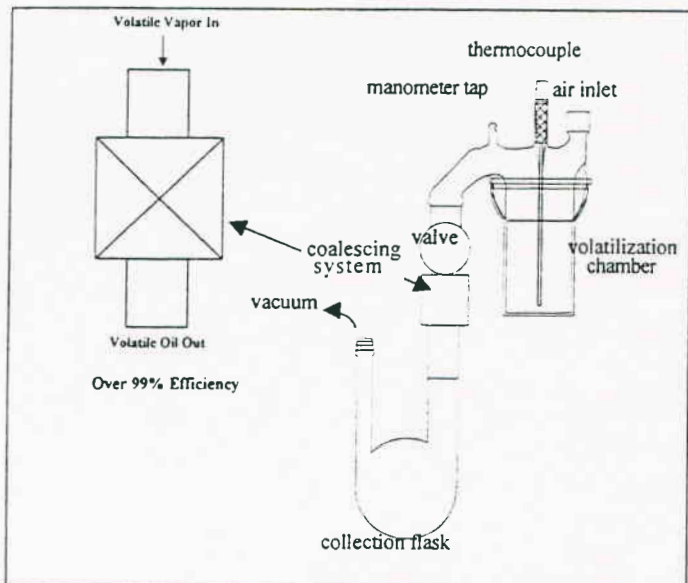
Figure 4: The new bulk volatility test set-up

orifice. Temperature signals from the J-type thermocouple are sent to a control console specially designed to house an internal transformer which conditions the output voltage to the noble metal heater of the volatilization chamber. The PID control of the control console is finely tuned to heat the oil from room temperature to 250° C in about 12 minutes.
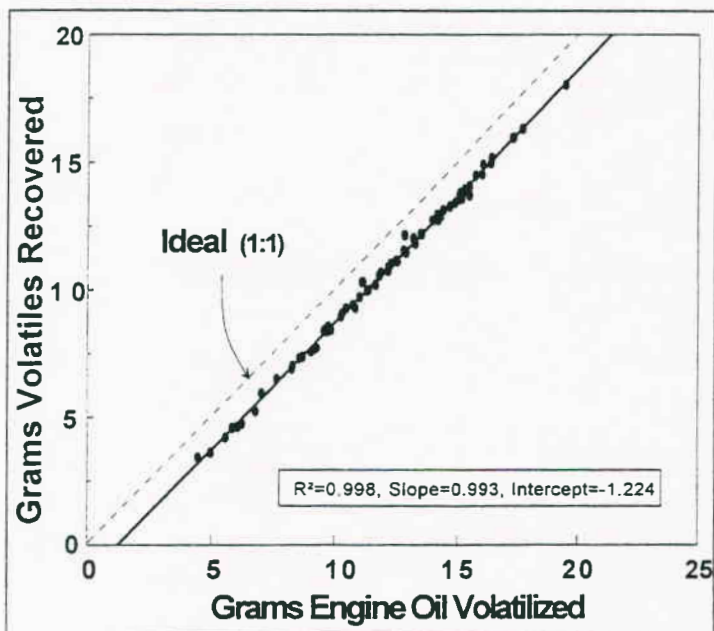

Figure 5: Generation and recovery of volatiles.

Volatile Collection - The bulk volatility test has a specially designed coalescing system placed just ahead of the collection flask (see Figure 4). The design of the coalescer gives an efficiency of about 99.5 percent recovery of the volatiles as shown in Figure 5 in which the amount of engine oil volatilized is compared with the amount recovered for an arbitrarily selected group of 67 oils analyzed in the prototype equipment.

At all levels of volatilization the recovery is essentially complete as shown by the slope of close to unity and the linear and closely packed data points. The constant difference of 1.22 grams between the amount volatilized and that collected is interesting. Since it is unlikely that the data points would be closely packed and at a ratio of 1:1 (unity slope) if oil vapor

were passing through the system, the most reasonably explanation is that the unaccounted weight of 1.22 grams of oil is consistently trapped on the surfaces of the coalescing system. An effort to wash and weigh the coalescing system showed that this was a reasonable explanation.

Technique - A tared, clean volatilization chamber is filled with 65.00 ± 0.05 grams of test oil. The bulk volatility test apparatus is then set up according to the sketch shown in Figure 4. With the valve closed completely, the vacuum pump is turned on to allow it to warm up and stabilize for 10 to 15 minutes. During the latter portion of this time, the control console is turned on to heat the volatilization flask to 250° C.

When the test oil reaches temperature, a timer is started for one hour and the valve is simultaneously but carefully opened to bring the vacuum pressure inside the volatilization chamber to 20 mm H_2O as indicated by an inclined manometer. The valve is adjusted, as needed, to keep the vacuum pressure at 20 ± 0.5 mm H_2O throughout the duration of the test.

At the end of 60 minutes ± 5 seconds, the vacuum and heat source are turned off. Following a 20-minute cool down period, both the volatility chamber and the collector flask are weighed to obtain percent loss and percent recovery respectively.

RESULTS

With the bulk volatility test apparatus design in hand, it became of interest to reestablish correlation with the Noack volatility test and determine repeatability. The opportunity came to participate unofficially in a Noack round-robin conducted in September 1995.[15] Nine laboratories officially participated in the round-robin. This paper presents data collected from that round-robin.

ENGINE OILS TESTED - The round-robin oils were five fully-formulated passenger-car engine oils. Other information such as viscosity/grade or additive package types was
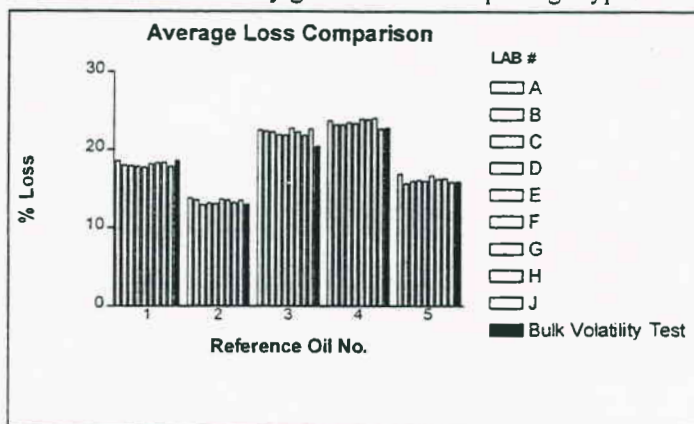

Figure 6: Comparison of volatilization data obtained on the Noack volatility test and the bulk volatility test.

unknown. Each laboratory had made five analyses with each oil and the same approach was made using the new method. A comparison of the average results from the nine laboratories and those from the authors' laboratory is shown in Figure 6.

DATA - Overall, the bulk volatility test and the Noack volatility test correlated reasonably well as shown in Figure 7. The Coefficient of Determination, R^2, equaled 0.964, slope was 0.892, and Y-intercept was 1.47%. Observation showed that for Oils 1, 2, 4, and 5, the loss values found with the bulk volatility method were within three percent of the round-robin
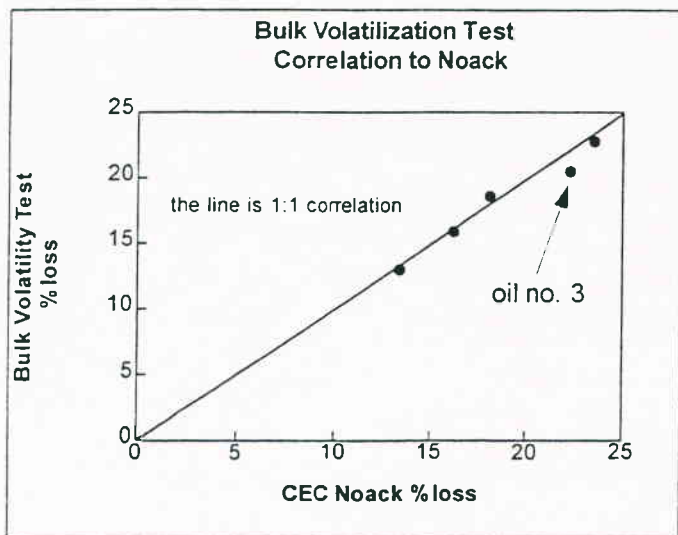
Figure 6: Volatility correlation chart between the Noack and the bulk volatility tests.

average which used the Wood's Metal Noack. Oil 3, however, was markedly lower (8.3%) than the reported average Noack value of the Round Robin and a question was raised regarding identity or contamination of the sample. This remains a question for future investigation.

If Oil 3 is removed from the data set, the Coefficient of Determination improves to R^2=0.986, the slope improves to 0.967, and the y-intercept improves to 0.374.

The repeatability data is also of interest (see Table 1). For oils 1, 2, and 4, repeatability was better than the average of the other nine labs. Oil 5 was calculated to be only slightly higher than the other labs. Oil 3 again stood out by giving considerably poorer repeatability than the Noack labs.

Oil No.	Viscosity cSt	Standard Deviation	
		Avg. of Noack Labs ASTM Round-Robin	Bulk Volatility Test
1	10.16	0.40	0.06
2	14.38	0.26	0.11
3	10.46	0.26	1.34
4	10.66	0.67	0.30
5	11.78	0.27	0.56

Table 1: Standard Deviations of participating labs in comparison to the bulk volatility test.

FUTURE WORK

The information gathered thus far on the bulk volatility test is encouraging. The authors feel the repeatability of this test can be improved even further with additional studies.

The bulk volatility test with its ability to yield a sizable sample of the volatilized material provides the opportunity of generating a deeper understanding of the effect of oil additives on the components of volatilization.

CONCLUSION

This paper has presented an instrumental technique that can provide information on volatility loss closely similar to the Noack Evaporation Test without the potential toxicity and hazards of using Wood's Metal. Moreover, the latter test provides for the collection of virtually all volatiles and provides opportunity to study these volatiles for important characteristics including the presence of exhaust catalyst contaminating compounds.

ACKNOWLEDGMENTS

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