Report to
Emissions System Compatibility Improvement Team (ESCIT)
On
Study of Extended-Time Phosphorus Emission Index (PEI) Values at 165°C

A Report by the ESCIT Task Group

Summary: Using a Matrix of eight oils with ZDDP products from the four major manufacturers and taxi-fleet field data, PEI(165) values obtained at 16, 32, 48 and 64 hours indicate that past engine and fleet correlation with PEI(165) at 16 hours is significantly reduced at longer exposure times. Different formulations of ZDDP in the Matrix Oils studied show different responses to extended PEI test periods.

Background

Two areas of interest developed from past studies of the phosphorus emission tendencies of engine oils using the Phosphorus Emission Index (PEI).

The first related to how well the PEI technique correlated with engine oil phosphorus emissions and catalyst deposition in engine and field tests. This has been answered in a series of studies beginning in 2004 **[Ref. 1-11]**.

More recent interest has been in regard to the amount of phosphorus emitted from engine oil with extended time in the PEI test and whether such time extension affects the aforementioned correlation established between engine and field tests at 165°C and 16 hours.

A Task Group was formed by ESCIT[1] to investigate this more recent interest.

(For reference in this report, the bench test instrument and essential components used in determining PEI are shown in Appendix 1.)

Composition of Task Group

Nine members of ESCIT volunteered to form the Task Group including the Sponsor of the PEI bench test as Chairman/Secretary. These individuals and their affiliations are shown in Table 1.

Appendix 2 presents the Task Group's Scope and Objectives as modified and accepted in its first telephone conference of July 2, 2007.

Table 1 -- Composition of Task Group on Extended Time Study of PEI

Name	Company	Industry Segment
Brad Cosgrove	Chrysler	Automobiles
Joe Franklin	Intertek	Laboratory
Greg Guinther	Afton	Additives
Wangkan Lin	Infineum	Additives
Jim Linden	GM	Automobiles
Ron Romano	Ford	Automobiles
Ted Selby	Savant	Laboratory
Jerry Wang	Oronite	Additives
Lew Williams	Lubrizol	Additives

Source of Data and Funding Support

As developer of the PEI method, Savant Laboratories were asked to run the desired test Matrix. Funding of the effort was undertaken by the Alliance of Automobile Manufacturers and industrial sources of ZDDP.

Choice of Matrix Oils

Eight oils representing products from each of the four ZDDP manufacturers were chosen by the Task Group in their telephone conference of July 2, 2007.

JAMA Oils

Engine phosphorus emission studies by the Japanese Automobile Manufacturers Association (JAMA) produced six oils, analyzed by the PEI(165-16) protocol. Of these six, Oils #2, #4, and #5 were selected for the Matrix. Associated JAMA engine test data were available for all six oils and had previously been reported to ESCIT **[7]**.

[1] This Task Group was formed at the ESCIT meeting in Arlington, Virginia, on June 14, 2007.

Lubrizol Oils

The Lubrizol Corporation was asked for two oils that formed the basis of a 2006 Lubrizol-Ford taxi-fleet test **[3].** Lubrizol identified one of the oils as having low impact on the catalyst while the other was indicated as being a more conventional formulation. These oils had also formed part of an earlier studies of PEI correlation **[3,4]**.

Afton Oils

Afton offered two oils both of which had been run in their ACT engine test protocol, one of which, FT-33, had been a part of an earlier Afton-Ford-Delphi fleet test in 2002 **[1]**. This fleet test used several oils – three of which were made available to first test PEI correlation **[2]**. However, it was noted by the Afton representative that Oil FT-33 was atypical in having no detergents and thus not representative of commercial engine oils.

Oronite Oil

The oil proposed by Oronite provided a ZDDP additive widely applied in the field and the Task Group agreed to incorporate this oil into the Matrix.

Final Oil Matrix

Table 2 shows the final set of Matrix Oils and sources established in the July 2nd telephone conference. By July 18th, all Matrix Oils were in hand to complete the work needed for the report to ESCIT on August 9, 2007.

Table 2 - Oils Selected for Test Matrix

Oil Source	Oil Code
JAMA	Oil #2
	Oil #4
	Oil #5
Lubrizol	'Low Impact'
	'Conventional'
Afton	FT-33
	443
Oronite	Low PEI

Associated Study of ICP Methods

Question of possible differences in phosphorus values between ASTM Method D 4951 and the more commonly used Method D 5185 were resolved by a three laboratory study of samples from the PEI test Matrix. No differences outside of test precision were shown.

Method D 4951 was shown as being preferable for precision in the analysis of oils from the PEI method according to ASTM observations stated in the method.

PEI Test Protocol

The Task Group selected PEI test time intervals of 16, 32, 48 and 64 hours. In this report the term PEI(165-T) will be used to generically designate the PEI tests at different hours whereas the specific number of hours will replace 'T' in more exact references.

Four Selby-Noack instruments were available for use in this study. A 10-gram co-volatile of phosphorus-free SN75 was used in all PEI analyses as practiced in the PEI(165-16) protocol. Operators established inlet air flow rate and position of air entry in the reaction flask (see Appendix 1) each day and adjusted it to a standard test condition used in Noack tests.

Limited Supporting Engine Data

Information on five of the Matrix Oils including fleet test data on the Lubrizol oils **[3]** and engine dynamometer phosphorus emission data plus Estimated IIIG Phosphorus Volatility data on the JAMA oils is presently available. However, no engine test data applicable across all eight Matrix Oils is presently in hand.

Requests have been made of the suppliers of the Matrix Oils for IIIG data.

However, the present lack of engine data was not expected to hinder gathering pertinent information regarding the cross-correlation of the results from PEI(165-T) analyses at the time intervals selected by the Task Group.

Results

Comparison of Data – Table 3 shows results of the present study.

Green highlighted information for the oils in Rows 1, 2, 3, 4, 6, 8, 9 and 11 show results obtained on the present samples of the eight Matrix Oils. The heavily outlined areas encompass the data requested of the Task Group and conducted under the funding offered.

Remaining green highlighted data are values obtained by Savant Labs to test the repeatability and consistency of the data obtained in these new operational protocols as well as adding relevant data to the Matrix.

The blue-highlighted information on the oils in Rows 4, 5, 7, 8 and 10 show the identity and PEI(165-T) values for oils previously sent to the Savant Laboratories well before the initiation of the study (some sent as long as a year or more ago).

In some cases, a number of analyses were run on a given oil sample over this time as part of earlier Savant studies and give evidence of the repeatability that can be obtained depending on the kind of ZDDP contained in the engine oil.

Table 3 -- ESCIT Extended PEI Testing Matrix

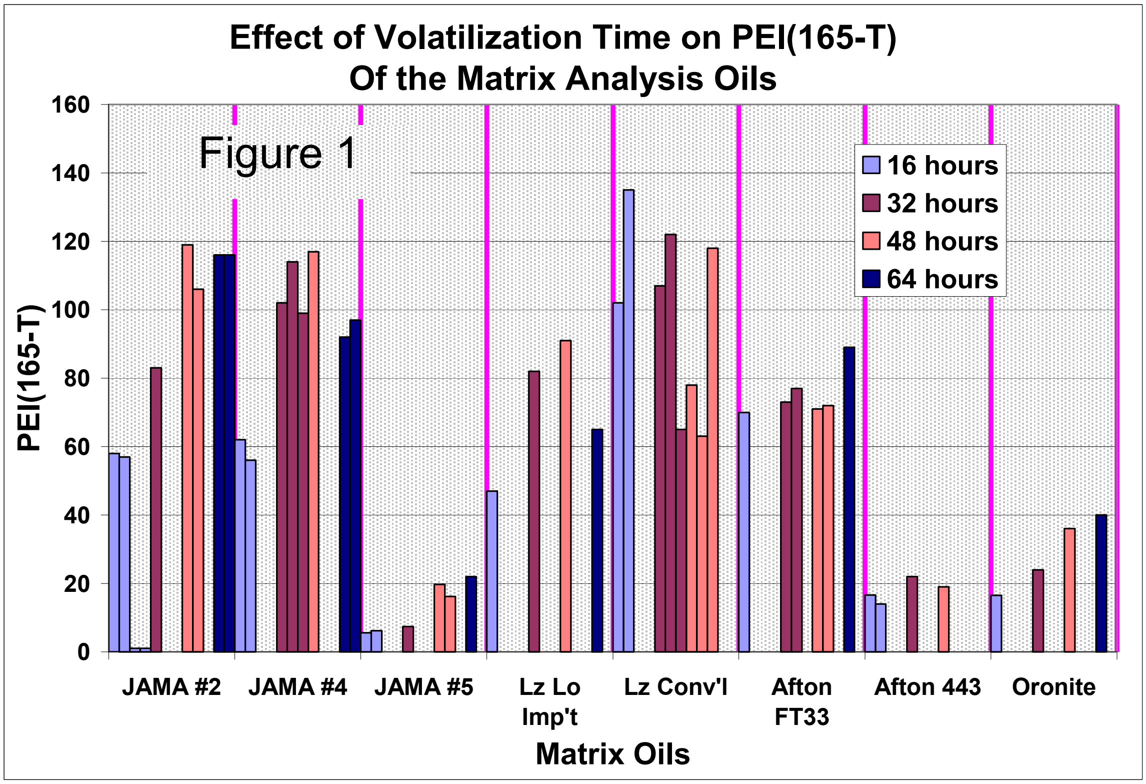
Oil Source		Oil ID	Oil Code (if any)	PEI @ 165ºC			
				16 hrs.	32 hrs.	48 hrs.	64 hrs.
JAMA	1	Oil 2		58	83	119	116
				57		106	116
	2	Oil 4		62	102	117	92
				56	114		
					99		
	3	Oil 5		5.6	7.4	19.7	
				6.2		16.2	22
Lubrizol	4	'Low Impact'	OS183034F	52	74	106	88
				52			
				51			
				46			
				52			
		'Low Impact'	OS183034F	47	82	91	65
	5	'Conventional'	OS018699A	105		148	
				98			
				98			
				97			
				95			
	6	'Conventional'	OS184699C	102	107	78	
				135	122	63	
					65	118	
Afton	7	FT-33	RBR-10358A-01	70			
	8	FT-33	RBR-10358A-02		77	72	
		FT-33	RBR-10358A-02	70	73	71	89
	9	443	RBR-10443-L-02	16.6	22	19	
				14.0			
Oronite	10	Low PEI	OR96906 TK151119	15.0			
	11	Low PEI		16.5	24	36	40

PEI values obtained on similar or same oils submitted earlier

Extended time PEI(165-T) analyses | Requested analyses

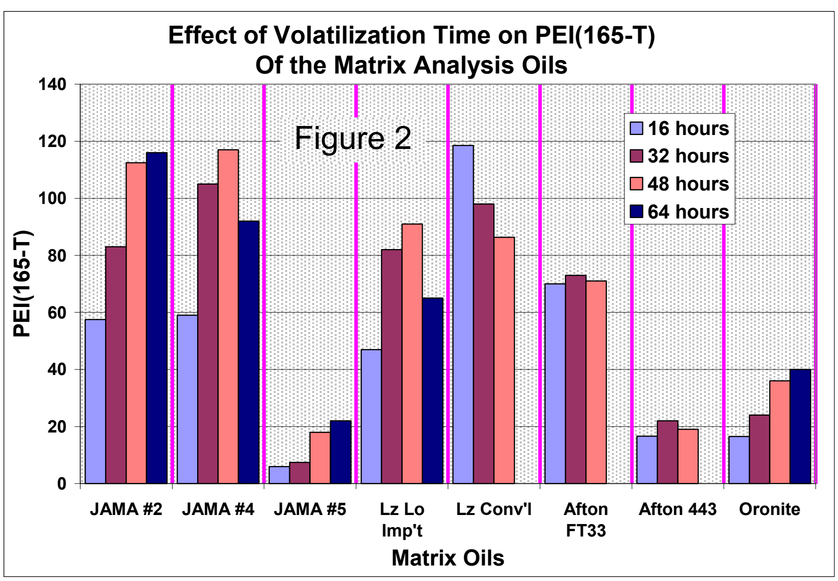
Effects of Increased Volatilization Time

Figure 1 is a bar chart of the PEI(165-T) values obtained at the four time intervals of Table 3. The chart shows that at a given exposure (for example, 32 hours), some of the Matrix Oils result in greater repeatability in the amount of volatilized phosphorus than others. Similarly, the bar chart indicates that the progression of PEI values with extended time is more regular with some of the oils than with others.



The values also show that a decrease in PEI(165-T) values can occur for some of the Matrix Oils and that in other of these oils the PEI(165-T) is essentially constant with increasing time of exposure.

PEI values previously shown in Figure 1 and Table 3 are averaged and plotted in Figure 2 for each Matrix Oil at each time interval.



Discussion

General

The study has produced several interesting pieces of information regarding phosphorus volatility which are covered in Appendix 3 regarding observations from the work of the Savant Laboratories as the information was gathered for this Task Group study.

Repeatability

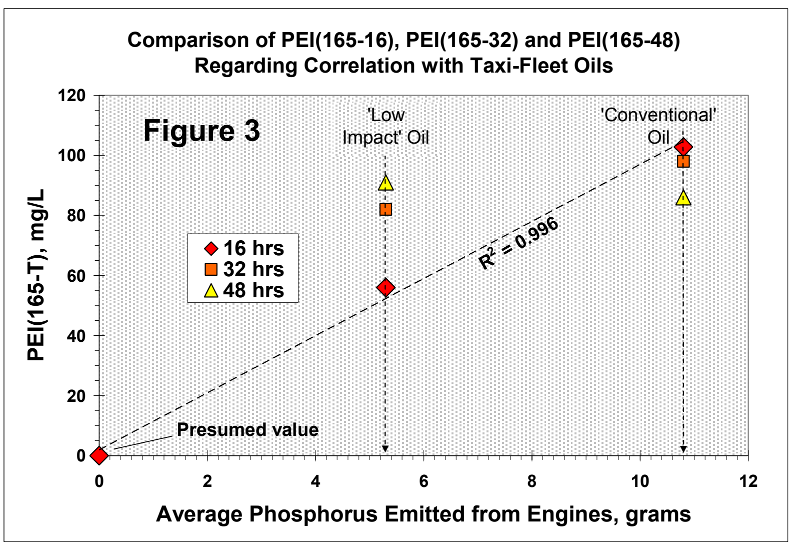
Table 3 shows the relatively good repeatability of the PEI protocol at 165°C and 16 hours with some, but not all, of the Matrix Oils. Such repeatability permits greater confidence in knowing when other factors such as significant variation in form and effects of ZDDP decomposition products are involved.

Present Exposure Time and ZDDP Decomposition Effects

Even without the availability of engine test data of the same type for each of the Matrix Oils, conclusions regarding extending the applicability of the PEI(165-16) correlation values can be made.

Figure 2 shows that within the repeatability and correlation already shown for PEI(165-16) **[1-11]**, variation among the PEI(165-T) values of the Matrix Oils with exposure time reduces correlation when compared to the values of PEI(165-16).

For example, as shown in Figure 3, the correlation previously found with PEI(165-16) values with the ‘Low Impact’ and ‘Conventional’ taxi-fleet test oils is essentially lost with increasing time of volatility exposure as noted by the inversion of PEI(165-32) and PEI(165-48) values for the two oils compared to the PEI(165-16) values for the same oils.



Conclusions

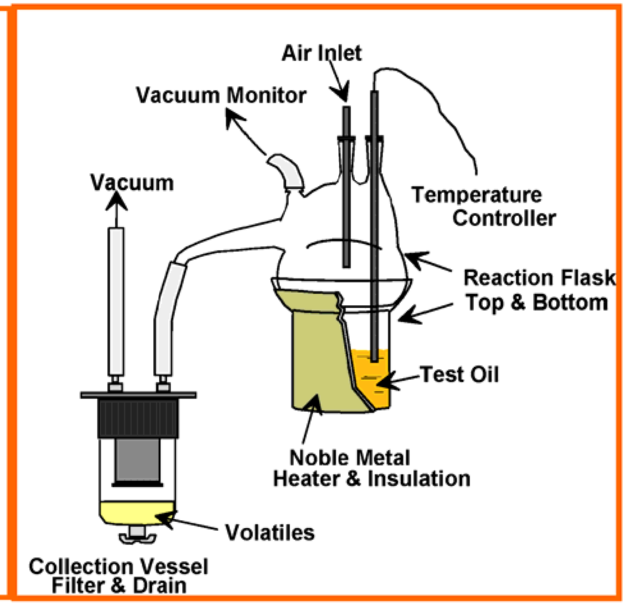
At this time, the data indicate that the level of correlation of PEI(165-16) with field and engine tests is reduced with increased time of exposure. Thus, the PEI(165-T) test length of 16 hours remains the best choice for engine dynamometer and field test correlation.

Respectfully Submitted to ESCIT

By Task Group Chair

Ted Selby

Appendix 1
Instrument and Important Components Used in Determining Phosphorus Emission Index



Appendix 2

Scope and Objectives as Modified in July 2nd Phone Conference

Scope

The Scope of the Phosphorus Emissions Bench Test Task Group is to determine the optimum test length for the PEI(165) protocol to obtain best correlation with field and engine tests using engine oils containing ZDDP products from all four manufacturers.

The Scope of the Task Group also includes the setting of Objectives.

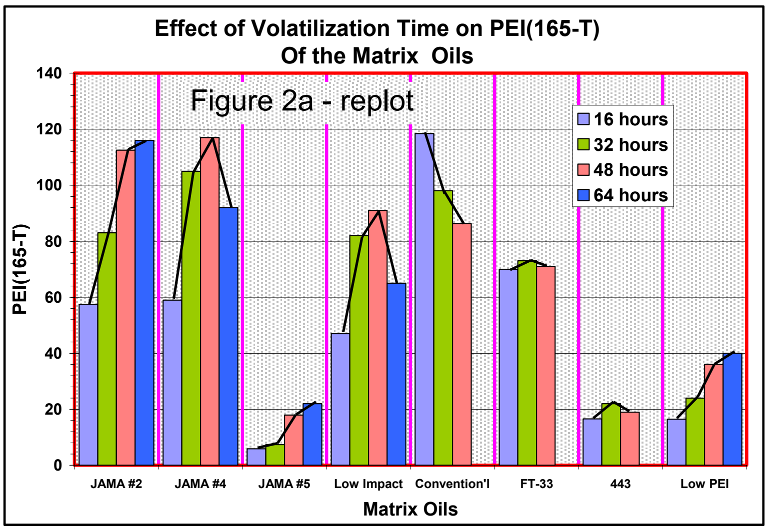
Objectives

1. To select a Matrix of engine oils containing ZDDP products from each manufacturer. The engine oils should have associated engine dynamometer or field tests directly or indirectly related to phosphorus emission.
2. To determine, through designed bench studies utilizing the Selby-Noack instrument, the optimum test length that will provide best correlation with data from taxi fleet and engine data.
3. To report the data gathered to the parent ESCIT group with recommendations regarding a subsequent round robin conducted under the auspices of ASTM Committee D02 and its Subcommittee B, Section 7.

Appendix 3
Observations of PEI Test Developer and Sponsor Concerning the Task Group Study Results Including October 4th ESCIT Meeting Report and Discussion

General

The Task Group study showed that increasing the time of volatilization had considerably different effects on the various Matrix Oils in regard to their phosphorous volatilities. This is shown in the modified replot of Figure 2 from the Task Group report (accordingly renamed Figure 2a). The heavy black lines connect the values of PEI at the indicated exposure times for each of the eight Matrix oils evaluated in the study. As stated, it is apparent that there are considerable differences among these oils in how they respond to the three or four conditions of the extended time PEI test at 165°C.



It seemed of technical value to understand more fully the causes of changes in PEI(165-T) values with increased volatilization time. The following observations, reasoning, and tentative conclusions resulted.

Variation in Volatility of Phosphorus from ZDDP Decomposition Products

Phosphorus-Containing Volatiles – Not surprisingly, considering the complexity and differences of various ZDDPs, as part of the PEI studies, phosphorus-containing volatile products have been found to vary depending on the decomposition chemistry of different ZDDPs at the temperature of exposure **[12-16]**.

The data of Figure 2a first of all clearly show the similarities and differences of response of the several forms of ZDDP submitted in the oils for the Matrix study previously noted:

1. Two different regimes of overall response are shown exemplified by oils identified as JAMA #2, #4, Low Impact, Conventional, and FT-33 having generally higher levels of PEI(165-16) phosphorus volatility compared to the oils identified as JAMA #5, 443, and Low PEI. (The ZDDPs in the oils identified as Low Impact, 443, and Low PEI have been suggested as more advanced ZDDP technology for the given manufacturer.)
2. Oils identified as JAMA #5 and Low PEI show similar low initial levels of phosphorus volatility which increases slowly toward a plateau.
3. The oils identified as FT-33 and 443 show little or no change in volatility after 16 hours of exposure although there is considerable reduction in phosphorus volatility in Oil 443 compared to Oil FT-33

4. The oil identified as JAMA #2 is similar to Oils JAMA #5 and Low PEI except the former shows much larger increases in phosphorus volatility with increasing exposure time in reaching toward a plateau.
5. The oils identified as JAMA #4 and Low Impact both show the interesting response of first showing large increase of phosphorus volatility with increasing exposure time followed by a substantial decrease in phosphorus volatility from 48 to 64 hours.
6. Perhaps most interesting of the eight oils is the oil identified as Conventional. This oil shows marked phosphorus decrease after the first PEI value at 16 hours.

Each of these phosphorus volatility patterns resulting from the reported time-exposure study have an explanation – some more obvious than others. However, some may have more than one rational explanation in which case similarly interesting PEI studies can be applied and coupled with other analytical techniques to determine the facts.

Explanations of Phosphorus Volatility Responses

General – The premises for the following explanations of the above phosphorus volatility responses of the eight Matrix oils are:

- **a.** The PEI is a repeatable test technique[2],
- **b.** The PEI protocol discriminates well at various levels of phosphorus volatility, and,
- **c.** Correlation of the PEI protocol with engine and field tests has been amply demonstrated.

Specific Explanations of Phosphorus Volatility Patterns – Essentially four patterns have been identified in the sequence of test at 165°C:

1. No change in phosphorus volatility level after 16 hours of exposure to PEI protocol at 165°C,
2. Increasing levels of phosphorus volatility tending to equilibrium,
3. Increasing levels of phosphorus volatility to a maximum followed by a significant decrease in phosphorus volatility, and .
4. Significantly decreasing levels of phosphorus volatility.

Pattern 1

This is the simplest response to the PEI protocol and is explained by the completion of all phosphorus volatilization at 165°C in 16 hours after which there is neither further phosphorus generation nor any loss of phosphorus already trapped in the volatiles Collection Vessel of the test instrument shown in Appendix 1.

Pattern 2

In this pattern, phosphorus volatiles continue to be released after 16 hours of exposure to 165°C at significant but diminishing rates. At the relatively high levels of phosphorus volatility produced, it is not presently known whether there is any re-volatilization or untrapped phosphorus volatiles. If there are, they are very minor in comparison.

Pattern 3

Phosphorus volatiles continue to be released after 16 hours of exposure to 165°C. A certain significant amount of these phosphorus-containing components have a more volatile character and perhaps less solvency in the co-volatilized oil. The loss of these latter phosphorus volatiles from the Collection Vessel with time ultimately is evident as a decrease in overall phosphorus volatility at sufficiently extended time.

[2] Several sources **[7,8,9,17]** as well as data contained in the Task Group report to ESCIT have demonstrated repeatability of the PEI(165-16) protocol.

Pattern 4

Maximum volatilized phosphorus is produced early in the PEI protocol consisting of a large amount of considerably more volatile phosphorus-containing components. The amount of phosphorus volatiles measured continually decreases from 16 hours on and seems to trend toward an ultimately constant level representing the minor presence of less volatile phosphorus components.

Discussion

Correlation Considerations – With the results of the completed extended-time PEI study, it is very interesting to note that PEI correlation with the field and engine emission data is best at the lowest time of PEI determination so far investigated – 16 hours. This result is obtained despite the fact that the study has shown that considerably larger levels of phosphorus are subsequently volatilized from some of the Matrix oils.

Moreover, with the so-called 'Conventional' oil studied, the amount of phosphorus volatilized decreased with increasing time from 16 hours on. Thus, correlation was found good only at this most limited time of PEI exposure.

It may be that, instead of extended time, less time than 16 hours at 165°C may improve correlation.

***Significance of Correlation at 16 Hours* –** The fact that correlation was reduced with exposure extended beyond 16 hours is an important finding from the study. It implies that phosphorus generation at 16 hours in the PEI protocol predicts the main influence of phosphorus on the catalyst and that later phosphorus generation in the test, although in some cases plentiful, does not have the same influence.

Thus, the PEI bench test seems to be providing information that may be important in the design of both engine oils and catalysts.

At least three explanations are reasonable:

1. The earliest forms of volatile phosphorus represented by the 16-hour PEI data are most damaging to the catalyst,
2. The effect of all phosphorus volatiles produced is greatest earlier in catalyst life when there are more sites for adherence. This is represented by the 16-hour data.
3. Particularly, in the field but also in engine tests, the oil is changed regularly and only the phosphorous emissions produced in the earlier period of its use are the emissions producing the catalyst degradation[3].

In brief, these three explanations involve 1) earlier and different forms of volatile phosphorus, 2) competition of any volatile phosphorus for adsorption sites on the catalyst during catalyst life, and 3) oil change affecting the amount, and perhaps type, of volatile phosphorus. It very well may be that the correct explanation lies in a combination of the three explanations.

***Degree of Phosphorus Volatility* –** The degree of volatility of different phosphorus-containing materials generated by the decomposition of ZDDP is also a consideration when discussing the results of the extended-time study.

Not surprisingly, considering the complexity and differences of various ZDDPs, as part of the PEI studies, phosphorus-containing volatile products have been found to vary depending on the decomposition chemistry of different ZDDPs at the temperature of exposure **[12-16]**. Three of the Matrix oils show a decrease in PEI, two after extended time intervals of 48-hours and the third immediately after 16 hours.

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[3] This was suggested by Dr. Dalrene Uy of Ford Motor Co. at the October 4th ESCIT meeting.

To investigate this occurrence, a very recent experiment by the PEI Test Developer showed that significant phosphorus could be found in the upper coalescing filter chamber above the Collecting Vessel (see picture of instrument in Appendix 1). This demonstrated that more highly volatile phosphorus-containing components can be produced by ZDDP decomposition and that these more volatile components continue to volatilize from the essentially ambient temperature Collecting Vessel over extended time.

Such loss of more volatile forms of decomposition of certain ZDDPs from the Collection Vessel might have been anticipated from the fact that phosphorus-containing components of ZDDP decomposition were found volatile down to Reactor Flask temperatures of 120°C **[4,5]**.

Others have also noted that all phosphorus in the volatile components of decomposed ZDDP may not be caught in the collection vessel. This was discussed in a relatively recent ESCIT meeting **[17]**. Thought has been given to increasing the efficiency of phosphorus collection. In the case of extended periods of exposure to volatilizing conditions, such as in this study, increased efficiency of phosphorus collection would be helpful in appraising the total phosphorus volatility.

An interesting question is related to what these more volatile phosphorus-containing decomposition products are and how they can be more efficiently trapped in the Collection Vessel and identified. Obviously, as shown by the combination of the PEI data on the 'Conventional' oil in Figure 2a and the correlation of PEI with the taxi-fleet data associated with this Matrix oil, such highly volatile decomposition products seem very efficient in degrading the catalyst.

Phosphorus Retention Index

The residual oil left in the Reactor Flask (see Appendix 1) after test is also a source of information regarding volatilized phosphorus. As part of the PEI protocol, phosphorus in the residual oil is also measured and a parameter called the Phosphorus Retention Index (PRI) is calculated in the same manner as the PEI.

As observed in the aforementioned ESCIT report **[17],** it is a more demanding phosphorus analysis because of so-called 'matrix' effects caused by the presence of polymeric materials in both the fresh and residual oils, both of whose phosphorus values are used to determine the PRI.

However, despite the comparative imprecision, the combination of the PEI and PRI in the present study has been found very useful. This was particularly the case with those Matrix Oils exhibiting a decrease in PEI values from one test period to the next.

Need for a Correlative Bench Test

There are obviously a number of factors involved in the choices of ZDDP chemistry to serve the engine effectively in regard to wear and oxidation control and with phosphorus volatility levels commensurate with maintaining exhaust catalyst life. Other additives used in formulating engine oils may also affect phosphorus volatility control; certainly their absence has been shown to increase phosphorus deposition.

The unexpected finding that phosphorus volatility is essentially independent of either oil volatility or initial phosphorus concentration was through the application of a bench test that later showed good correlation with engine and fleet tests **[2]**.

Engine tests, while important to establish the data desired for correlation, have become too expensive, imprecise, inflexible, and logistics-dependent to make effective long-term standards of evaluation. Properly designed, knowingly applied, and relevantly correlated bench tests can buttress and extend the application of technical information from engine studies and provide a bridge to the next engine test level of desired lubricant performance.

References

[1] Johnson, M.D., et. al., **“Effects of Engine Oil Formulation Variables on Exhaust Emission in Taxi Fleet Service”,** SAE Powertrain & Fluid Systems, Paper 2002-01-2680, San Diego, California, October 21-24, 2002. Published by SAE, *Lubricants,* SP-1722, pp.53-71, 2002.

– Ford, Afton, Delphi study of phosphorus catalyst deposits

[2] Savant report to members of ILSAC on correlation of PEI with Ford, Afton, and Delphi taxi-fleet test **[1]**, November 4, 2004.

– Good correlation with three fleet test oils that had been made available for this comparison.

[3] Lubrizol, **“Effect of ZDP Chemistry on Three-Way Catalysts (TWC) Phosphorus Exposure and Emissions Performance”,** Presentation at ESCIT meeting on August 8, 2006, Ann Arbor, Michigan.

– Lubrizol-Ford taxi-fleet study of effect of two ZDDP chemistries on catalysts.

[4] Savant, **“Field Correlation with Bench Test Studies of Engine Oil Phosphorus Volatility”,** Presentation at ESCIT meeting on September 28, 2006, Romulus, Michigan.

– Correlation of PEI with Lubrizol taxi-fleet study.

[5] Savant, **“Phosphorus Emission Index Studies of, and Correlation with, Taxi Fleet Engine Oils”,** Presentation at ESCIT meeting on December 12, 2006, Pontiac, Michigan.

– Correlation of PEI with taxi-fleet studies of **[1]** and **[2]**.

[6] Chevron Oronite, **ESCIT Objectives, IIIG-PEI Precision and Correlation,** Presentation at ESCIT meeting on December 12, 2006, Pontiac, Michigan.

– Good precision and correlation of PEI with Sequence IIIG and other information.

[7] Chevron Oronite, **“Phosphorus Volatility – Chevron Oronite Perspective”,** Presentation at ESCIT meeting on February 22, 2007, San Antonio, Texas.

– Good correlation of PEI with field, IIIG, and other data led to Oronite choice of PEI from among other options.

[8] Afton, **“Support for a Phosphorus Volatility Specification in GF-5”,** Presentation at ESCIT meeting on February 22, 2007, Detroit, Michigan.

– Good repeatability and IIIG correlation led to support of PEI(165-16).

[9] Infineum, **“Phosphorus Volatility Test for GF-5”,** Presentation at ESCIT meeting on June 14, 2007, Arlington, Virginia (Washington, D.C.).

– PEI(165-16) correlates well with IIIG phosphorus retention.

[10] JAMA, **“JAMA Emission Compatibility Work”,** Presentation at ESCIT meeting on August 8, 2006, Ann Arbor, Michigan.

– Special engine dynamometer studies of phosphorus emission and correlation with Estimated IIIG Volatility.

[11] Task Group Chairman’s E-mailed letter of August 15, 2007 comparing PEI(165-16) data on all six JAMA oils evaluated and reported earlier (see **[10]**).

– Good correlation ($R^2 = 0.83$) when all six oils were evaluated. Also showed evidence of a consistent bench test.

[12] Bosch, R.J., et. al., **"Continued Studies of the Causes of Engine Oil Phosphorus Volatility",** SAE Powertrain and Fluid Conference, San Antonio, Texas, October 27, 2005

[13] Savant, **"Studies of PEI of Fresh Engine Oil at Both Ring Belt and Lower Temperatures",** ILSAC-ESCIT Meeting, Livonia, Michigan, December 15, 2005.

[14] Selby, T.W., **"Phosphorus Emission Index and Related NMR Studies of ZDDPs at Two Different Temperatures",** 15th International Colloquium Tribology, Ostfildern, Germany, January 18, 2006.

[15] Savant, **"Phosphorus Emission Index at Different Temperatures and Length of Exposure Time",** ILSAC-ESCIT Meeting, Auburn Hills, Michigan, February 28, 2006.

[16] Selby, T.W.**, "Studies of Engine Oil Phosphorus Volatilization Using Phosphorus Emission Index (PEI) Techniques",** Symposium on Automotive Lubricant Testing and Additive Development, Lake Buena Vista, Florida, December 3-5, 2006.

[17] ExxonMobil, **"ExxonMobil Phosphorus Volatility Studies – 1/22/07 Update",** Presentation at ESCIT meeting on February 22, 2007, Detroit, Michigan.