

**Subject:** GF-5 Emissions System Compatibility Improvement Team Meeting Minutes

Charlie Sherwood of Ford opened the meeting at 9:00 AM on 6/28/05 at Ford's facility in Livonia, MI.

1. A sign in sheet was circulated. See attachment 1.
2. The official member rooster was circulated for review and update. See attachment 2.
3. The agenda was reviewed and accepted as proposed. See attachment 3.
4. The team's charter was reviewed and slightly modified. See attachment 4.
5. Ted Selby gave a presentation on engine oil phosphorus and sulfur volatiles.
  - a. See attachment 5 for Ted's presentation.
  - b. Ted commented that having fleet tested oils with a range of ZDDPs and catalyst performance and phosphorus capture levels would be beneficial to correlate the PEI and SEI testing to actual field performance.
6. Doug Deckman gave a verbal presentation on work XOM has done on of various ZDDPs to measure volatility. Doug said he may be able to give a more detailed presentation in the future to the team.
7. Brent Schoffner reviewed the previous work done by the OPEST 2 team. See attachment 6 for Brent's presentation.
  - a. Sherwood asked SWRi if there is enough time to develop OPEST III into a test for GF-5. Brent responded that he is uncertain that there is a willingness by the industry to accept OPEST as a test.
  - b. Doug Ball made the point that with the 2007 emissions limits the catalysts efficiencies need to be 99% for NOx. Will any of these tests be capable of measuring that level of efficiencies?
  - c. Is there really enough time to do the work we need in time for GF-5?
  - d. Is anyone running testing that can be shared with the team and when will you have data available? The Team will pull together a spread sheet of the available data which might help shed light on emissions test selection and field testing to lab test correlation. AFTON, LZ and FORD indicated they are working on field trails and may have data to share in the future. We need a description of the oils run, the engines, test duty cycle, the catalyst/oxy sensor description and analysis conducted. Include work on low phos oils relative to engine durability. Request a dead line to respond if we have captured the correct parameters for the spread sheet of 3 weeks. Then once the spread sheet is complete, ask for data in 3 weeks. See attachment 8 for a first cut at the parameters we need to capture for the available data. Please add to the list and return your comments to [lawm@lubrizol.com](mailto:lawm@lubrizol.com).
  - e. Selby stated that the PEI is showing promise but needs correlation work to the field.
8. The team was asked to send to LAWm ([lawm@lubrizol.com](mailto:lawm@lubrizol.com)) references to papers that relate emissions system durability.
9. Doug Ball expressed concern that it would be difficult to develop a test to measure the impact of sulfur on catalyst durability.
  - a. Sherwood to determine from ILSAC and JAMA if there is a push to control sulfur below the current GF-4 levels in GF-5.
  - b. With engine oil consumption dropping is sulfur an issue?
10. Status Report to ILSAC/OIL on 6/29/05. The team discussed what to report to ILSAC/Oil. See the discussion below.
  - a. Clarify ILSAC's position on their willingness to drop the elemental limits for GF-5 if an adequate performance based test is developed.

- b. The Team is concerned that we do not have enough time to progress a performance based test in time for GF-5. Can we meet the dead line of 1/1/07?
- c. We are running testing on current hardware. Is current hardware indicative of 2009 hardware?
- d. The Team recommends we develop a set of ref oils which can be used to establish correlation between the field and bench, chemical and dyno testing.
- e. Is it a safe assumption that if phos exits the engine it ends up on the catalyst and that phos on the catalyst is bad?
- f. Will there be any support for a performance based test verses elemental limits?
- g. ILSAC/OIL should continue work to establish elemental limits for GF-5 since the Team can not guarantee we will be able to develop a performance based test for GF-5.
- h. Determine what sulfur limits are needed for GF-5. Will sulfur limits be lowered from GF-4, dropped or remain the same? Why does ILSAC/JAMA feel we need a sulfur limit?
- i. Is the expectation that sulfur limits need to be reduced in GF-5?

11. See attachment 7 for the report to ILSAC/Oil.

Next meeting will be called after we work out the details of the spread sheet.

Lew Williams

**GF.5 Emissions system Compatibility Team  
June 28, 2005 Meeting**

<b>NAME</b>	<b>ADDRESS</b>	<b>PHONE/FAX/EMAIL/ MOBILE</b>
Ball, Douglas	Delphi MC: 485-220-070 1601 N. Averill Avenue Flint, MI 48556	810-257-5204 doug.ball@delphi.com
Bardasz, Ewa	Lubrizol 29400 Lakeland Blvd. Wickliffe, OH 44092	440.347.2445 <a href="mailto:eab@lubrizol.com">eab@lubrizol.com</a>
Deckman, Doug	Exxon Mobil	856-224-2658 doug.deckman@exxonmobil.com
Ellis, Scott	Southwest Research Institute 6220 Culebra Road San Antonio, TX 78238	210.522.3399 210.684.7530 <a href="mailto:scottiellis@swri.org">scottiellis@swri.org</a> 210.313.9924 (Mobile)
Engel, Chris	DaimlerChrysler, CIMS 482-00-13 800 Chrysler Drive Auburn Hills, MI 48326-2757	248.576.7487 248.576.7491 248.361.7067 (Mobile)
Fee, Darrell	Astaris 385 Marshall Avenue Webster Groves, MN 63119	314.674.2188 <a href="mailto:Darrell_C_fee@astaris.com">Darrell_C_fee@astaris.com</a>
Franklin, Joe	Perkin Elmer Automotive Research 5404 Bandera Rd San Antonio, TX 78238	210.523.4671 210.523.4694 210.863.0438 (mobile)

NAME	ADDRESS	PHONE/FAX/EMAIL/MOBILE
Guinther, Greg	Afton Chemical 500 Spring Street Richmond, VA 23118	804.788.5368  <a href="mailto:greg.guinther@aftonchemical.com">greg.guinther@aftonchemical.com</a>
Jensen, Ron	Ford Research	<a href="mailto:rjensen2@ford.com">rjensen2@ford.com</a> 313-594-9629
Klein, Rick	Chevron Oronite 143 Cady Center #226 Northville, MI 48167	248.380.0625  <a href="mailto:rmkl@chevrontexaco.com">rmkl@chevrontexaco.com</a>
Ritchr, Andrew	Infineum 1900 E. Linden Avenue Linden, NJ 07036	908.474.2092
Roby, Stephen	Chevron Oronite 100 Chevron Way Richmond, CA 94802	510.242.1273  <a href="mailto:stephenroby@chevrontexaco.com">stephenroby@chevrontexaco.com</a>
Selby, Ted	Savant Inc. 4800 James Savage Midland, MI 48640	989.496.2301 989.496.3438 <a href="mailto:tselby@savantgroup.com">tselby@savantgroup.com</a>
Shoffner, Brent	Southwest Research Institute 6220 Culebra Road San Antonio, TX 78238	210.922.6986  <a href="mailto:bshoffner@swri.org">bshoffner@swri.org</a> 210.260.3830 (Mobile)
Weber, Ben	Southwest Research Institute 6220 Culebra Road San Antonio, TX 78238	210.522.5911  <a href="mailto:bweber@swri.org">bweber@swri.org</a> .
Williams, Lewis	Lubrizol 29400 Lakeland Blvd. Wickliffe, OH 44092	440.347.1111 440.944.8182 <a href="mailto:lawm@lubrizol.com">lawm@lubrizol.com</a>

## ESCIT Official Members

---

---

- Alex Boffa / (alternate) Steve Roby – Oronite  
[abof@ChevronTexaco.com](mailto:abof@ChevronTexaco.com)  
(510) 242-5220  
fax: (510) 242-3173  
[StephenRoby@chevrontexaco.com](mailto:StephenRoby@chevrontexaco.com)
- Chris Engel - DaimlerChrysler  
[cae8@dcx.com](mailto:cae8@dcx.com)  
(248) 576-6938  
fax: (248) 576-7490
- Joe Franklin / (alternate) John Glaser – Perkin Elmer  
[joe.franklin@perkinelmer.com](mailto:joe.franklin@perkinelmer.com)  
(210) 523-4671  
mobile: (210) 863-0438  
[john.glaser@perkinelmer.com](mailto:john.glaser@perkinelmer.com)  
(210) 647-9459  
fax: (210) 523-4607
- Andrew Ritchie  
[Andrew.Ritchie@infineum.com](mailto:Andrew.Ritchie@infineum.com)  
(908) 474-2097
- Ron Jensen – Ford Research  
[rjensen2@ford.com](mailto:rjensen2@ford.com)  
(313) 594-9629  
fax: (313) 248-5368
- Ewa Bardasz / (alternate) Jack Kelly – Lubrizol  
[eab@lubrizol.com](mailto:eab@lubrizol.com)  
(440) 347-2445  
fax: (440) 347-4635  
[jack@lubrizol.com](mailto:jack@lubrizol.com)  
(440) 347-1638  
fax: (440) 347-1733
- Greg Guinther – Afton Chemical  
[Greg.guinther@aftonchemical.com](mailto:Greg.guinther@aftonchemical.com)  
(804) 788-5368  
fax: (804) 788-6243
- Ted Selby – Savant, Inc  
[tselby@savantgroup.com](mailto:tselby@savantgroup.com)  
(989) 496-2301  
fax: (517) 496-3438
- Scott Ellis / (alternate) Bill Buscher – SwRI  
[Scott.ellis@swri.org](mailto:Scott.ellis@swri.org)  
(210) 522-3399  
fax: (210) 522-7530  
[William.buscher@swri.org](mailto:William.buscher@swri.org)  
(210) 522-6802  
fax: (210) 684-7523
- Charlie Sherwood - Ford Motor Company  
[csherwoo@ford.com](mailto:csherwoo@ford.com)  
(734) 458-0988  
fax: (734) 523-3979
- Doug Deckman – ExxonMobil  
[Doug.deckman@exxonmobil.com](mailto:Doug.deckman@exxonmobil.com)  
(856) 224-2658
- Doug Ball – Delphi  
[doug.ball@delphi.com](mailto:doug.ball@delphi.com)  
(810) 257-5204

**AGENDA**  
**GF-5 Emissions System Compatibility Improvement Team**  
**June-tbd-2005**

---

---

1. Membership changes
2. Review and approval of charter (to be discussed at 4-14-05 ILSAC/OIL Committee meeting).
3. Round-table discussion/review of latest findings on Impact of Phosphorus on Emission Systems
  - PEI procedure and review of latest findings
  - Phosphorus depletion of used oil from Sequence tests and field trials
  - Chemical Limits
  - OPEST review and potential for GF-5
  - Other ideas
4. Round-table discussion of Impact of Sulfur on Emission Systems
  - Is there a need to control Sulfur for GF-5?
  - Is a sulfur limit required with the move to Group II & Group III base oils?
  - What bench/functional test(s) relate to the impact of sulfur on emissions system durability?
  - Chemical limits for GF-5
  - Fuel sulfur effects/limits
  - Other ideas
5. Backwards Compatibility
  - OEMs will need GF-5 to back-service all older vehicles
  - What concerns does the team have over backward compatibility?
  - What would be required to demonstrate backwards compatibility?
6. Follow-Up items
7. Next Meeting

**GF-5 Emissions System Compatibility Improvement Team**  
Updated 6/28/05

**CHARTER**

The charter of the GF-5 Emissions System Compatibility Improvement Team is to evaluate potential methods for determining the impact of GF-5 engine oil formulations on emission system function & durability. The focus will be the impact of phosphorus and sulfur on catalysts and oxygen sensors. The team will consider physical, bench, field and engine tests as an alternative to chemical limits. The Team will make a recommendation to the GF-5 ILSAC/Oil Committee by 1-1-2007.

# Engine Oil Phosphorus and Sulfur Volatiles



## Some Considerations on Phosphorus and Sulfur Volatiles from Engine Oil

Meeting of the Emission System  
Compatibility Improvement Team  
June 28, 2005

# Engine Oil Phosphorus and Sulfur Volatiles

## Background

Analysis of phosphorus volatilities (Pvol) of over 1,000 oils in the IOM Database in a specially designed Noack showed that Pvol is not related to oil volatility or initial phosphorus level when exposed at 250°C for one hour.

It was suggested that the most likely other sources for Pvol were the effects of other additives in the engine oil formulation and the chemistry of the ZDDP.

For comparison, this same study generated the Phosphorus Emission Index or PEI as a comparative measure of Pvol:  $PEI = \text{mg}_p/\text{Liter}$

Subsequent work showed that phosphorus volatility was, in fact, related to the presence of other additives in the engine oil formulation.

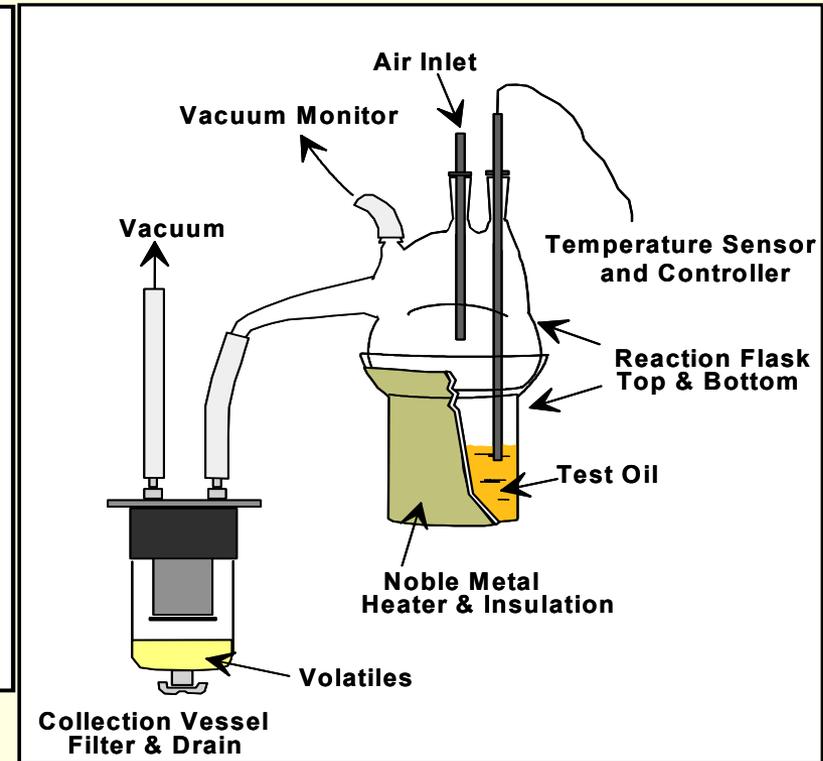
Moreover, limited data on one ZDDP showed correlation between PEI and catalyst degradation.

# Engine Oil Phosphorus and Sulfur Volatiles

For those who may not be familiar with the work, the instrument generating the PEI data is a special form of Noack that collects virtually all volatiles.



Selby-Noack instrument



# Engine Oil Phosphorus and Sulfur Volatiles

After showing that other additives do affect phosphorus volatility, work using both the special Noack and NMR turned to the role of ZDDP and its chemistry in a typical GF-3 engine oil techniques.

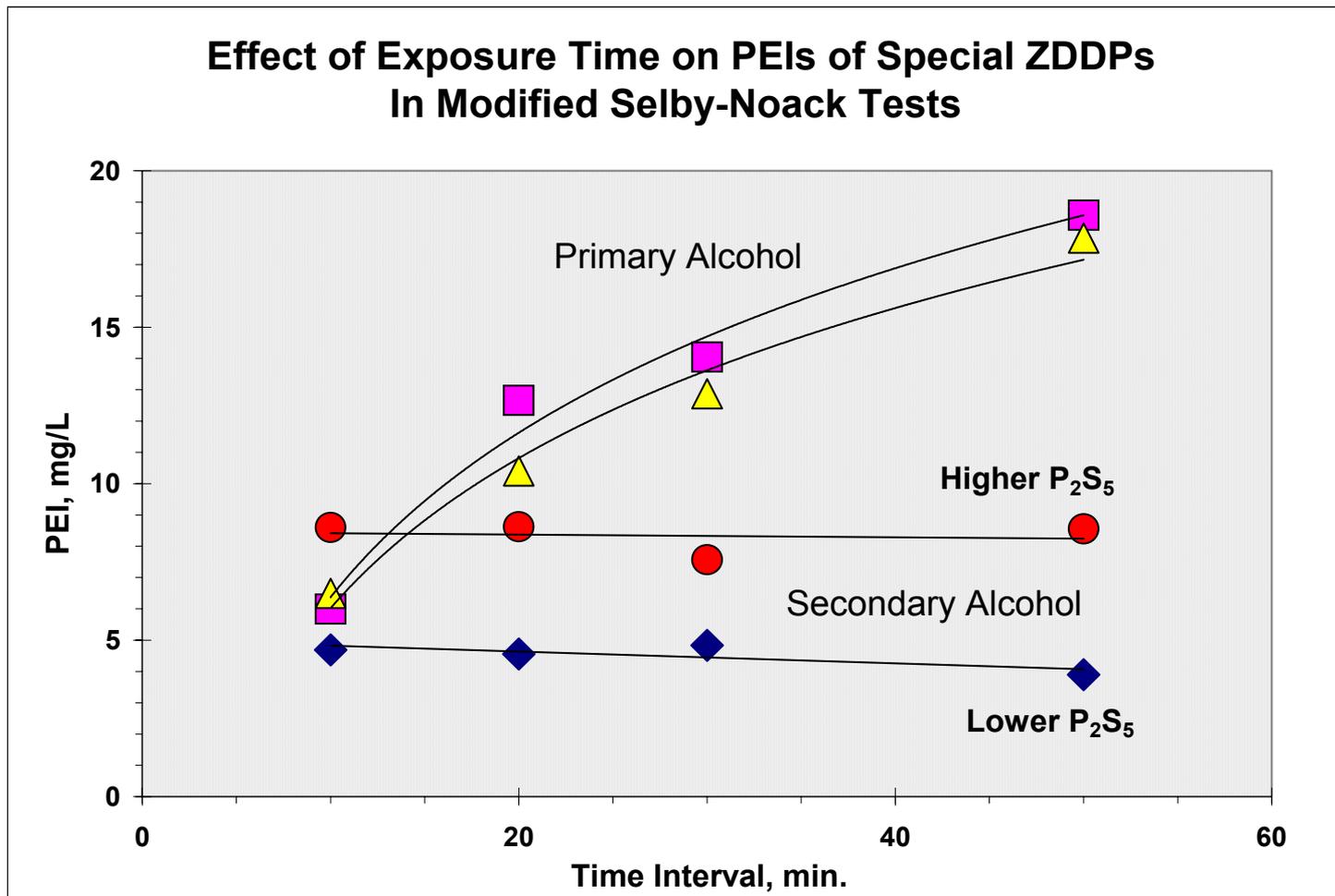
Among the studies was one that evaluated the progressive volatilization of phosphorus with time of exposure to the Noack temperature of 250°C.

Moreover, the study was made with both a primary and a secondary alcohol using two slightly different stoichiometries of  $P_2S_5$  – one higher, one lower.

This aspect of the study showed that phosphorus in the ZDDP made with a primary alcohol continued to volatilize over the course of the test and had higher values of PEI than a secondary alcohol.

# Engine Oil Phosphorus and Sulfur Volatiles

The PEI data show the differences in chemistry and formation of these ZDDPs.



# Engine Oil Phosphorus and Sulfur Volatiles

---

## Some considerations

Phosphorus volatility at 250°C is reflective of the temperatures of the piston ring belt. At lower temperatures phosphorus volatility would be considerably lower. Reasonably, the chemistry of ZDDP decomposition might also be different.

It is thus important to determine whether the much higher rate of phosphorus decomposition in the ring belt area is the driving mode behind phosphorus volatiles affecting the exhaust catalyst or whether lower temperature modes of ZDDP decomposition are most influential.

Fleet studies of ZDDP chemistry and catalyst effects would answer this.

# Engine Oil Phosphorus and Sulfur Volatiles

## Sulfur Volatility Studies

Sulfur in the engine oil as a component of the base oil or of the additive package, is also present in the volatiles and adversely affects emissions of NO<sub>x</sub>.

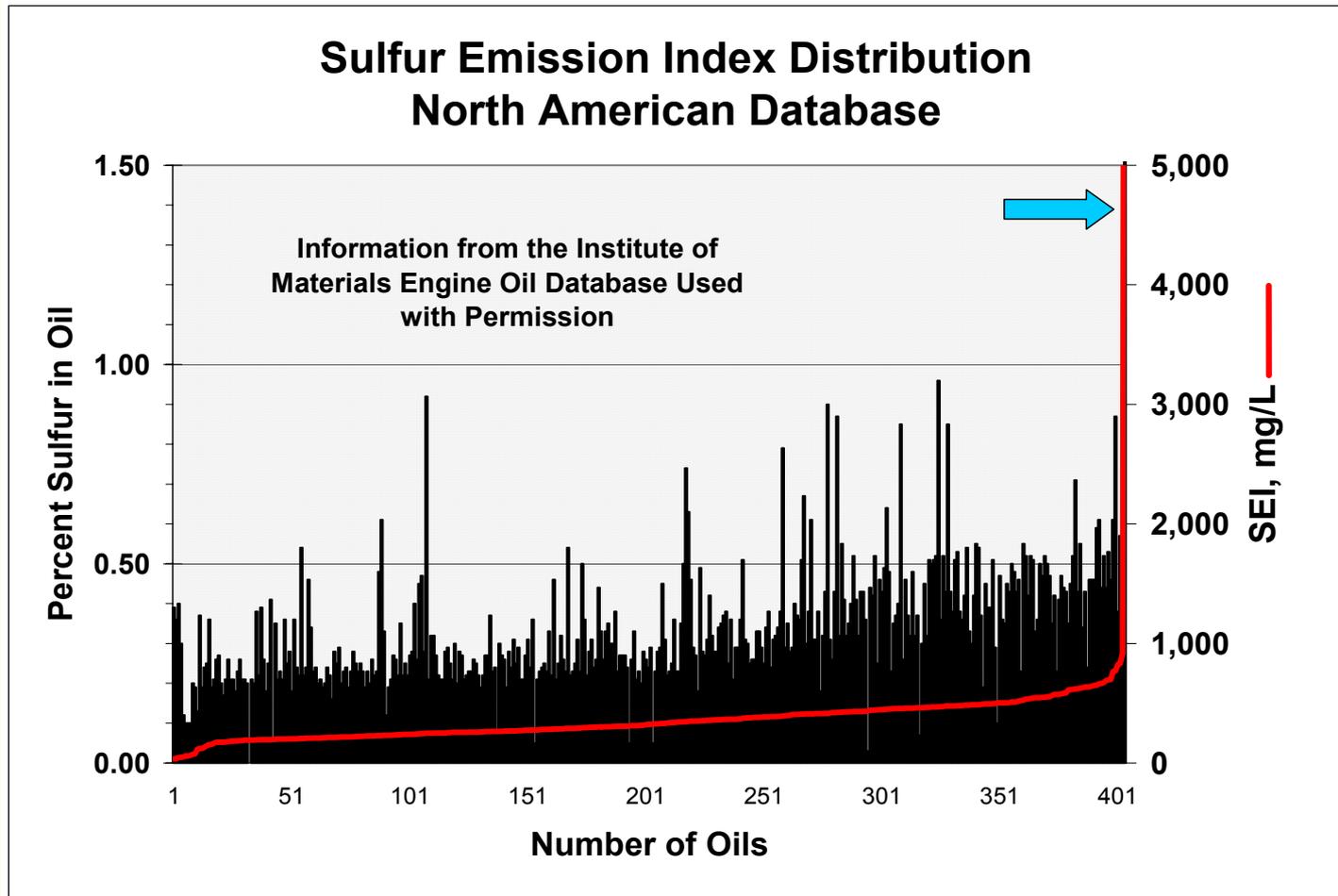
The special Noack instrument used in phosphorus volatility studies is similarly applicable to sulfur volatility and work has been recently reported including the use of the Sulfur Emission Index:

$$\text{SEI} = \text{mg}_S/\text{Liter}$$

As would be expected, SEI is related to some extent to the base oil and its volatility when the base oil carries sulfur-containing components of its own.

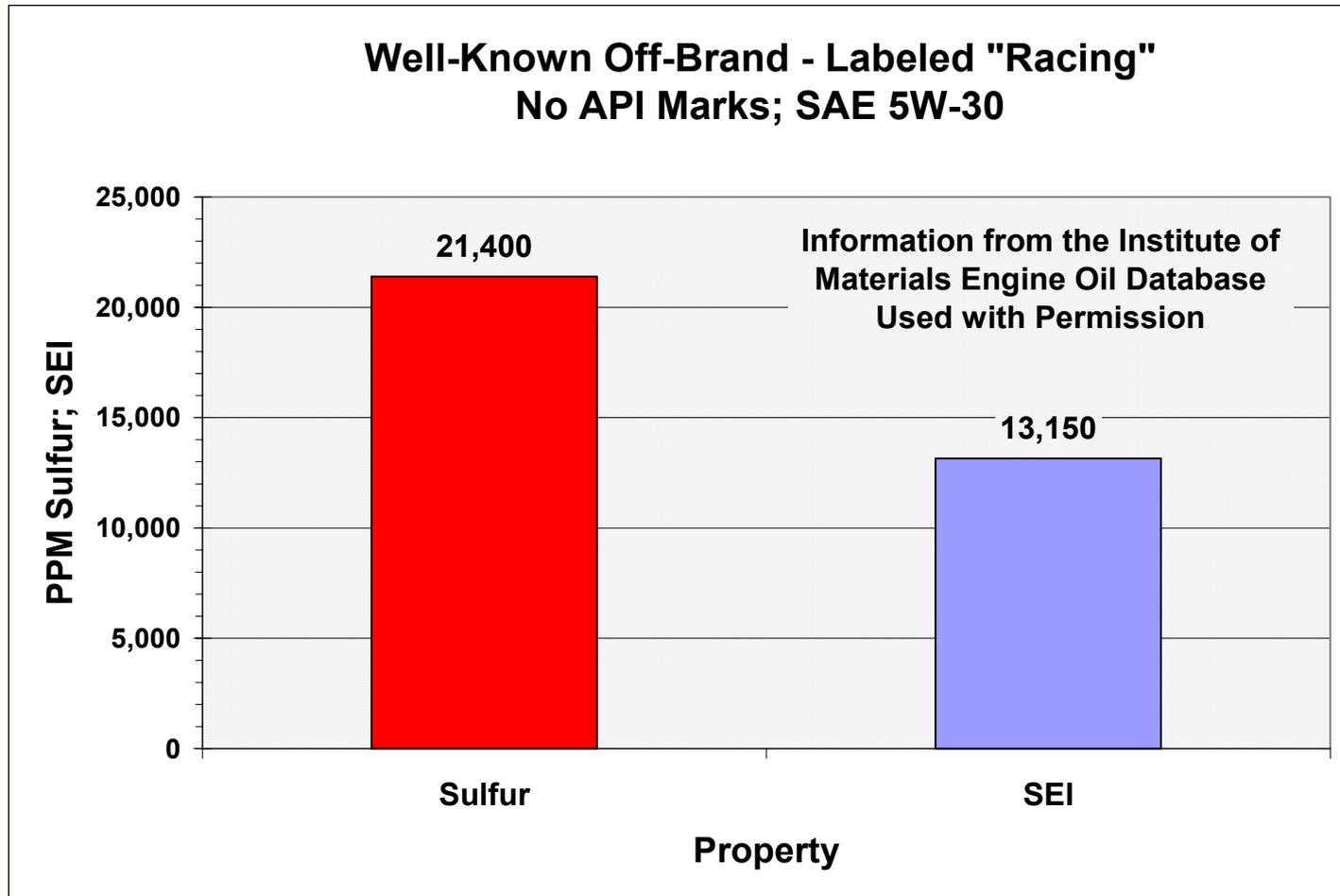
# Engine Oil Phosphorus and Sulfur Volatiles

The distribution curve for sulfur volatiles from North American oils is approximated by SEI with evident exceptions.



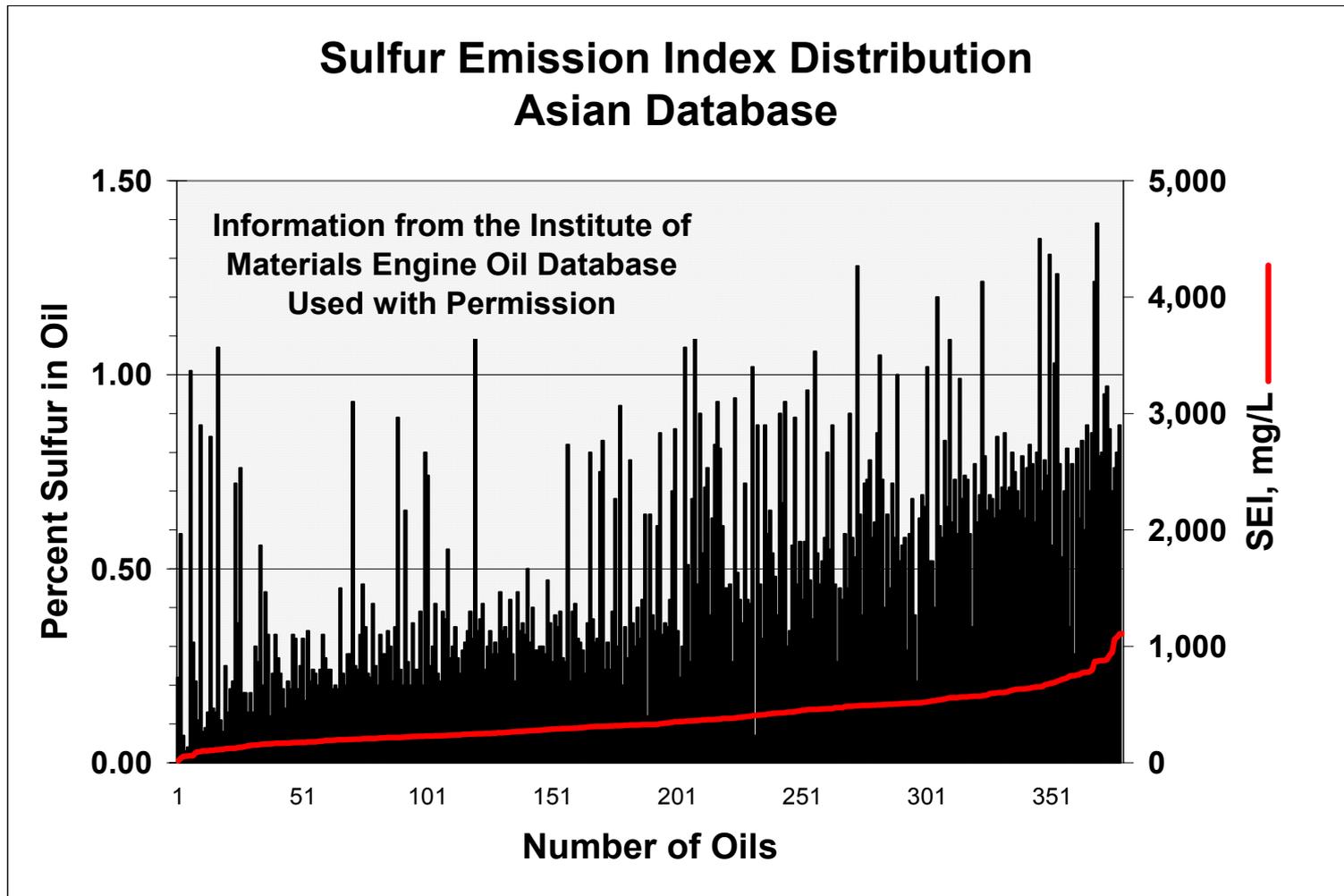
# Engine Oil Phosphorus and Sulfur Volatiles

An obvious exception with undoubtedly exceptional effects on emissions:



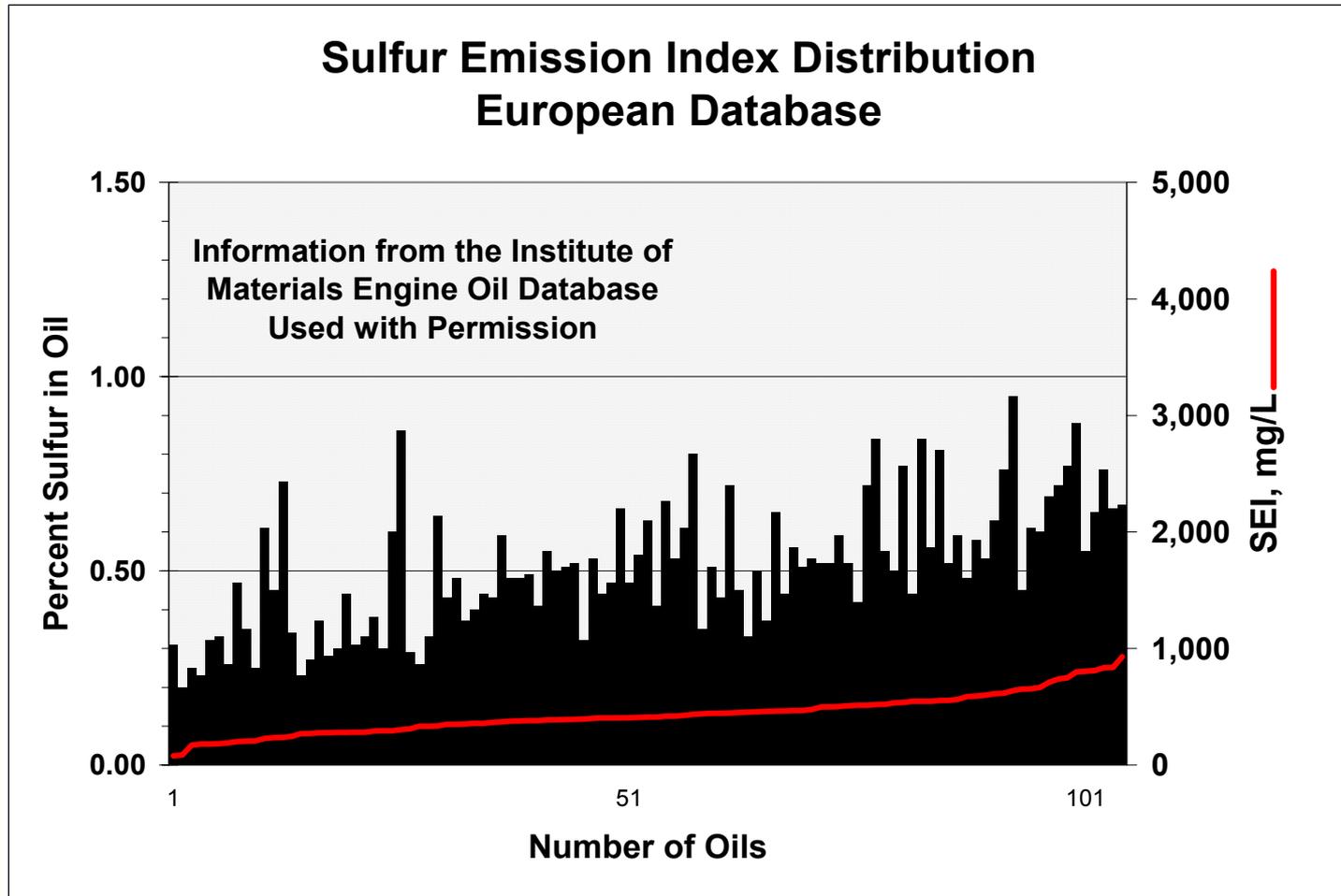
# Engine Oil Phosphorus and Sulfur Volatiles

The distribution curve for sulfur volatiles from Asian oils is approximated by SEI with more evident exceptions.



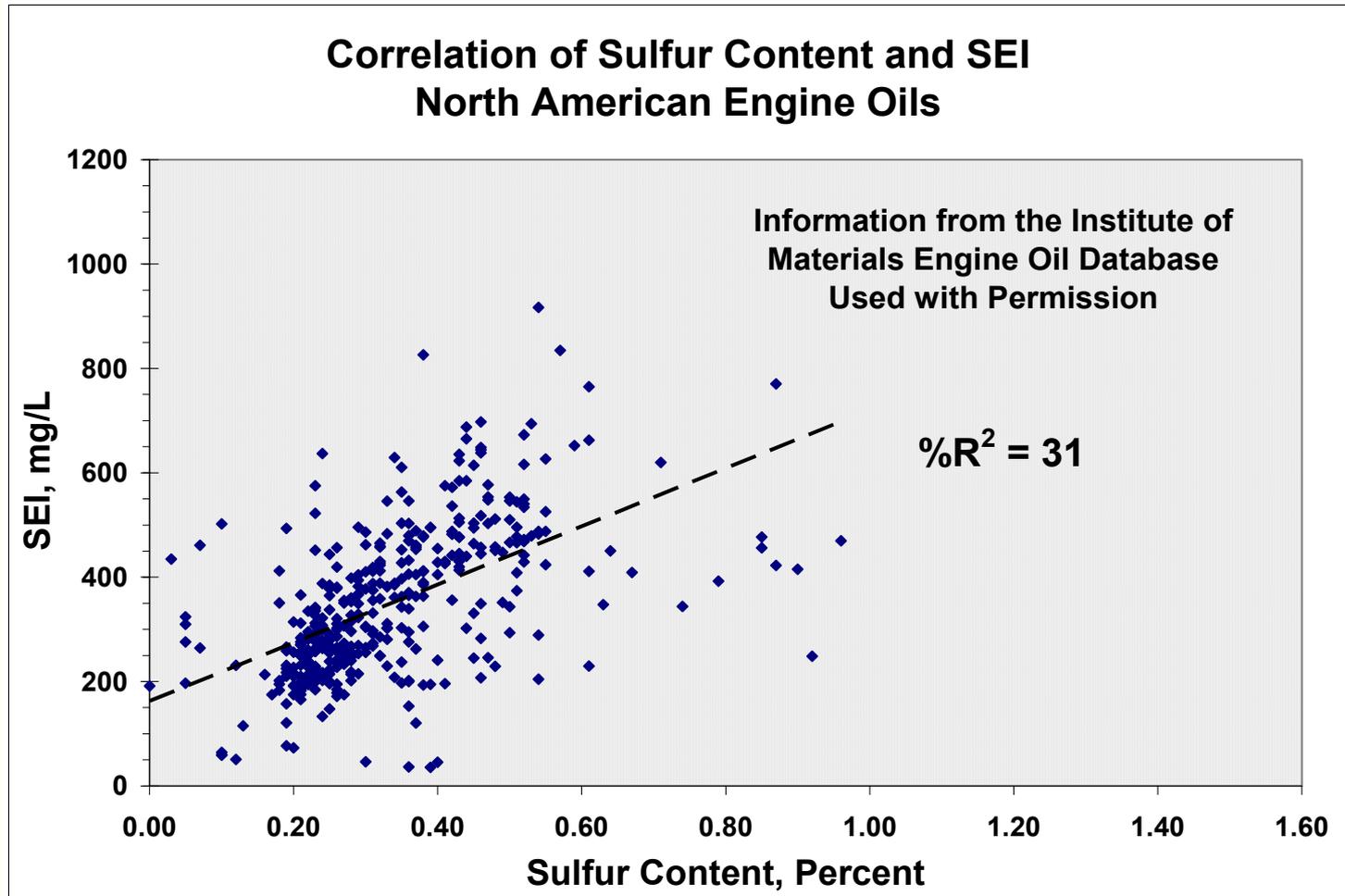
# Engine Oil Phosphorus and Sulfur Volatiles

The distribution curve for sulfur volatiles from European oils is also approximated by SEI with some exceptions.



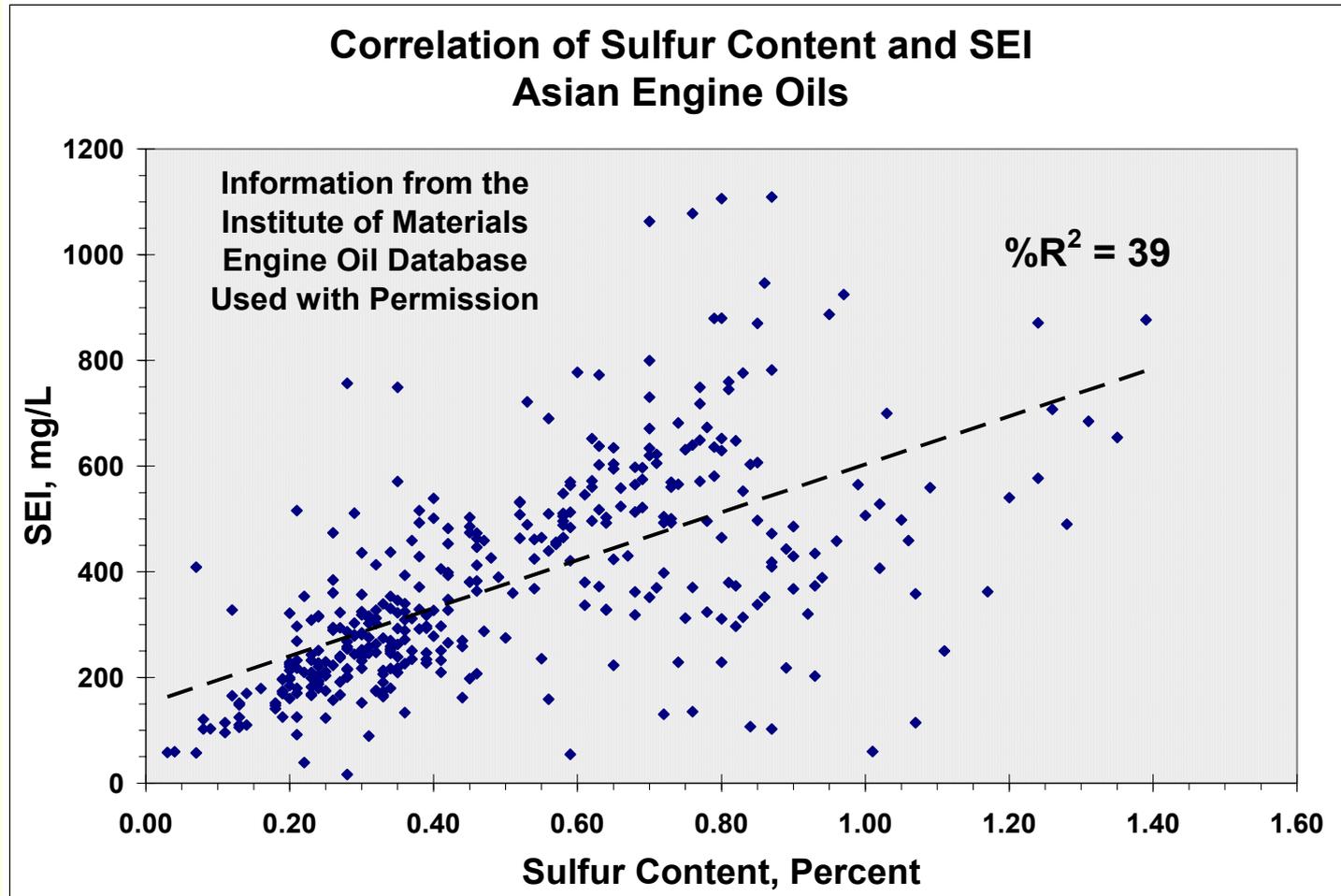
# Engine Oil Phosphorus and Sulfur Volatiles

A 31% correlation between SEI and sulfur content in North American oils leaves ~70% from other sources.



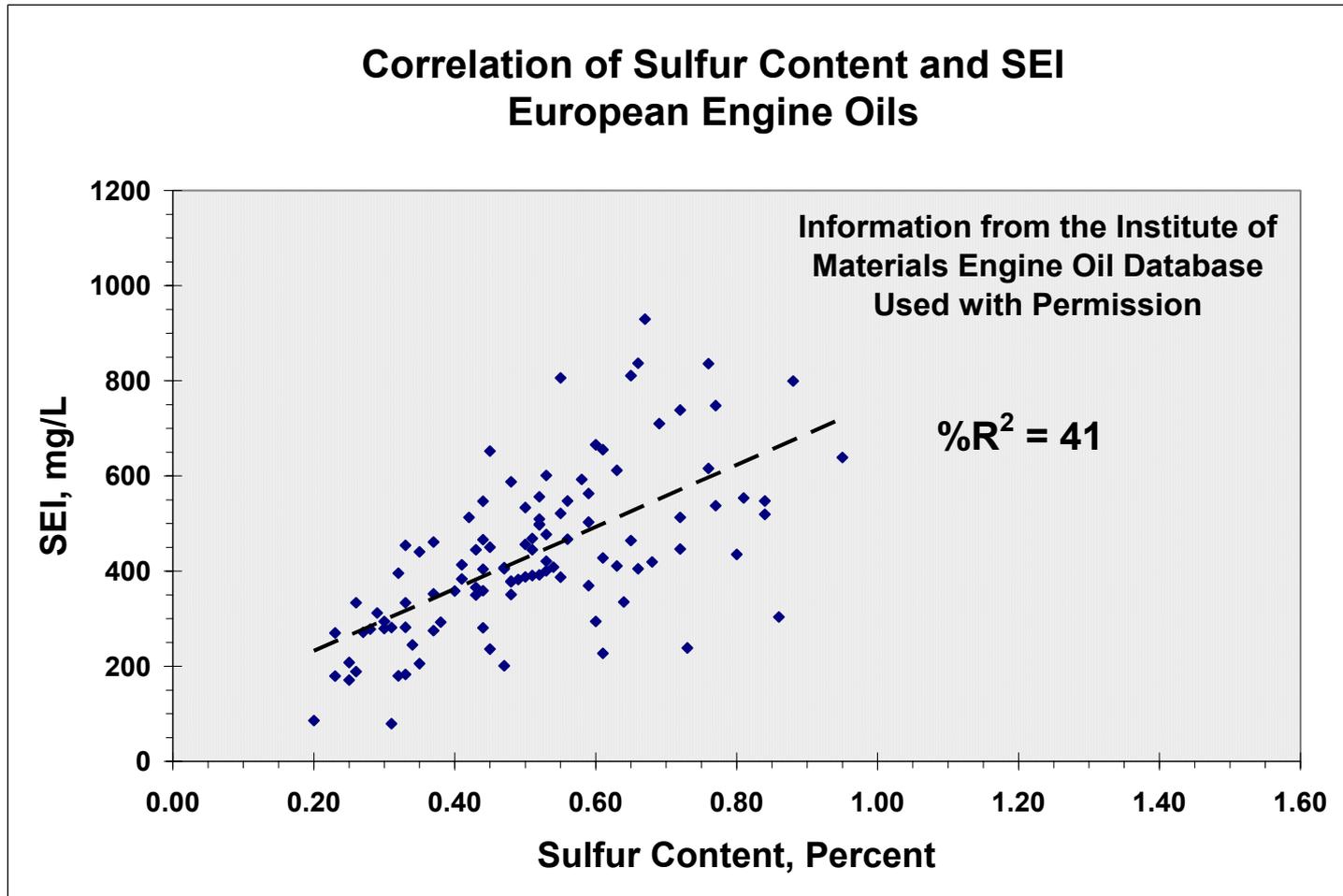
# Engine Oil Phosphorus and Sulfur Volatiles

Asian oils show a higher correlation of 39% likely because of higher levels of sulfur in some base oils.



# Engine Oil Phosphorus and Sulfur Volatiles

European oils show a correlation of 41% but in this case it may relate to greater additive uniformity.



# Engine Oil Phosphorus and Sulfur Volatiles

---

## Considerations

The SEI studies show that there is significant correlation with the percent sulfur in the initial engine oil.

However, the studies also indicate the degree to which sulfur volatility is influenced by other factors.

The data also suggest that there are combinations of base oils and additives that significantly depress sulfur volatility and others that enhance such volatility.

Much information remains to be generated from the IOM database such as degrees of correlation of SEI with sulfur-containing additives.

# Engine Oil Phosphorus and Sulfur Volatiles

---

## Conclusions

Measurement of the volatility of phosphorus and sulfur have shown the weakness of using fresh oil chemical concentration levels.

Phosphorus and Sulfur Emission Indexes have given information pertinent to development of control of these two chemical forms affecting exhaust emissions.

The fact that the present PEI and SEI data are produced by exposure of the engine oil to ring belt temperatures may generate a mode of ZDDP decomposition not occurring at lower temperatures.

Fleet study information on the correlation of higher temperature, more rapid phosphorus decomposition to catalyst degradation is important.

**SOUTHWEST RESEARCH INSTITUTE**™

6220 CULEBRA ROAD • POST OFFICE DRAWER 28510 • SAN ANTONIO, TEXAS 78228-0510, USA • WWW.SWRI.ORG

January 4, 2003

TO: ASTM Oil Protection of Emissions Systems Test (OPEST II) Task Force

SUBJECT: ASTM OPEST II – Completion of the Agreed Scope of Work

**I. BACKGROUND**

Four catalysts were each aged for 200 hours in the FOCAS™ Rig using the mild-thermal aging cycle with the engine oils noted in Table 1 on page 3. Cores from the aged catalysts were each tested four times in the SwRI synthetic gas reactor (SGR) using an enhanced stoichiometric perturbed light-off procedure developed to improve precision. A SwRI statistician analyzed the SGR T<sub>50</sub> light-off results and statistical differences with a 95 percent confidence level exist between Oil A and Oil B, and also between Oil 33 and Oil 34. Four catalysts were also selected at random and were preconditioned for 20 hours in the FOCAS™ Rig using the mild-thermal cycle. Two enhanced SGR tests were conducted on each one of these cores to compare preconditioned SGR data to the SGR results after 200 hours of aging.

SwRI recommended that sufficient aging hours had been performed and the ASTM OPEST II Task Force agreed with that recommendation at the OPEST II Task Force Meeting in Romulus, Michigan on May 15, 2002. The results of the program presented by SwRI at the subject meeting are included in the meeting minutes and posted on the Internet. Please refer to the link entitled "OPEST II Program Results - May 15, 2002" in the OPEST website:

<http://www.swri.org/opest/opest.htm>

At the meeting SwRI was assigned the following three remaining action items listed below. This report documents the completion of these action items

1. During the program the SwRI synthetic gas reactor (SGR) rig was improved to enhance precision. SwRI was tasked with determining the temperature at which the largest performance differences were observed for the tested catalysts, and reporting those results at the optimum temperature, with a full statistical analysis.
2. Obtain Proton-Induced X-ray Emissions (PIXE) results on the OPEST II and Ford samples.
3. Measure surface areas of OPEST II cores and field-aged catalysts supplied by the Ford Motor Company.



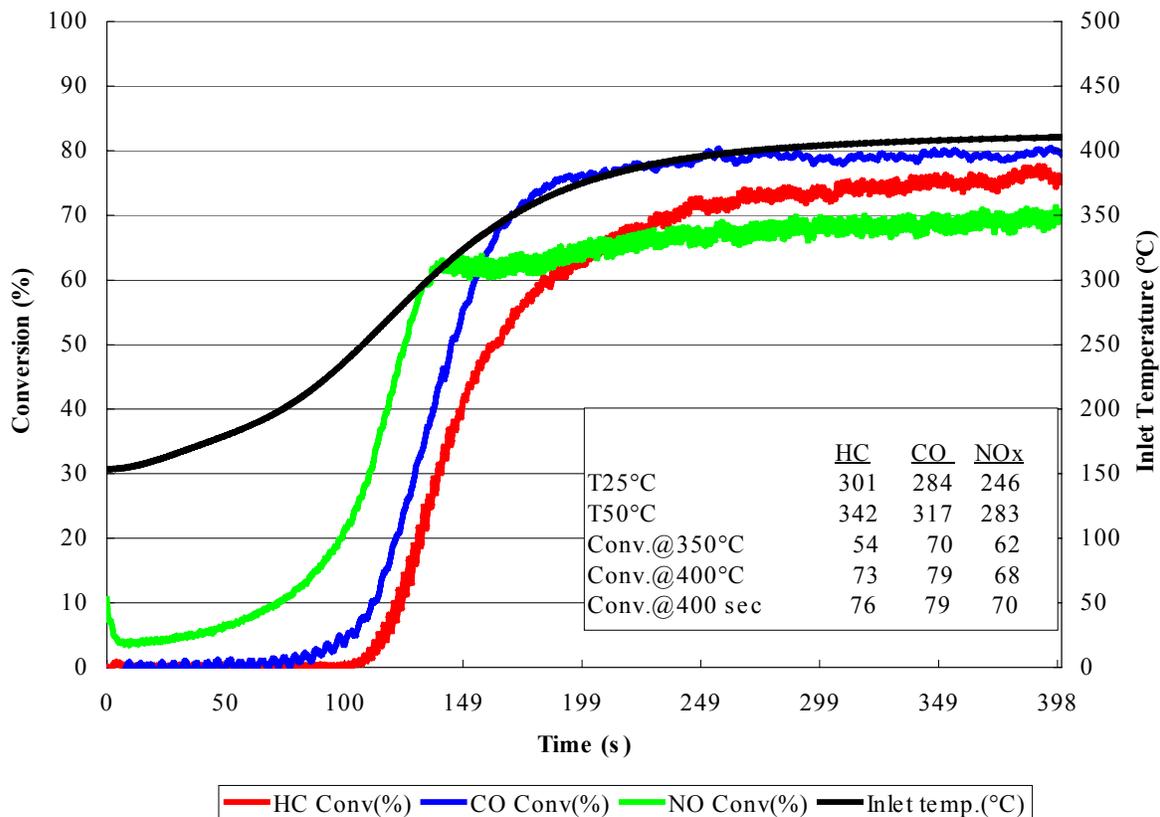
DETROIT, MICHIGAN (248) 353-2550 • HOUSTON, TEXAS (713) 977-1377 • WASHINGTON, DC (301) 881-0289

## II. RESULTS

### A. SGR Conversion Efficiency Temperature Study

For the reference core a graph of the SwRI SGR conversion efficiency and inlet temperature versus test time is shown in Figure 1. Data in this format were taken on the test cores in the OPEST II program. Prior to the SwRI SGR rig enhancement, conversion efficiency had been determined at 350°C and that temperature designation was used for the first OPEST II program core analyses. At the request of the ASTM OPEST II Task Force, SwRI determined the HC, CO, and NO conversion efficiency at temperatures of 250°C, 260°C, 270°C, 280°C, 290°C, and 300°C for the 200-hour aged cores from the OPEST II program data. The recalculated results for 290°C are shown in Appendix C.

**Figure 1 – Conversion Efficiency and Inlet Temperature Versus Test Time**



### Light-Off Test (Perturbation) - LP Core

The two questions to be answered for these data include:

1. Are the average efficiencies at Oil A different than at Oil B?
2. Are the average efficiencies at Oil 33 different than at Oil 34?

The full statistical analysis performed by Janet Buckingham of the SwRI Statistical Analysis Section is included as Appendix A. It appears that using 290°C or 300°C discriminate between the two oil groups (Questions #1 and #2) for all three emissions constituents. At the other conversion temperatures investigated (250°C, 260°C, 270°C, and 280°C) there was insufficient evidence to conclude that there was a difference between the two oil groups for some or all three of the emissions constituents.

In comparing the average HC, CO and NOx efficiencies between the appropriate oils, efficiencies calculated at 290°C and 300°C produced similar results. Thus, one is not more "optimal" than another. They both discriminate in the same fashion. The conversion efficiency of 290°C was selected for future testing. The conversion efficiencies for HC, CO, and NOx that were calculated from the OPEST II data at both 290°C and 350°C are given in a table in Appendix C and also compared graphically.

In addition another question was answered for the 290°C conversion efficiency results. Are the average efficiencies of Oil 33 different than of Oil B? An ANOVA was used to compare the average efficiency among the oils for HC and NOx. For HC, there was no significant difference in the average HC in comparing Oil 33 and Oil B. For NOx, there was a significant difference in the average NOx in comparing Oil 33 and Oil B. A nonparametric Kruskal-Wallis test was used to compare the medians of the CO efficiency. There was no significant difference in the median CO between Oil 33 and Oil B.

#### B. Proton-Induced X-Ray Emissions

The Proton-Induced X-Ray Emission (PIXE) results for the catalyst cores from the OPEST II evaluation program are shown in Table 1 and graphically in Appendix B. The material used for the PIXE evaluation was composed of a representative sample of the material from the cores tested in the OPEST II program. The PIXE results in weight percent are given in Table 1.

**Table 1 – PIXE Results from the OPEST II Cores**

Core	Oil	Detergent	P In the Oil	PIXE Analyses		
				P (wt%)	Zn (wt%)	Ca (wt%)
3	34	Ca/Mg	0.00%	0.000	0.009	0.261
20	B	Fully formulated	0.06%	0.952	0.825	0.315
13	33	No Detergent	0.10%	2.397	0.913	0.071
25	A	Fully formulated	0.11%	2.089	1.140	0.460
Ford CHNYT09	33	No Detergent	0.10%	6.536	0.855	0.093
Ford CHNYT06	34	Ca/Mg	0.00%	0.000	0.015	0.073

The amount of phosphorus in each test oil is listed in Table 1. Note that the PIXE result for phosphorus in core 13 (2.397 wt%) was greater than in core 25 (2.089 wt%) even though the phosphorus content in fresh Oil 33 (0.10%) was less than in Oil A (0.11%). Perhaps the detergent package in Oil A mitigated the phosphorus poisoning on core 25. Oil 33 had no detergent package.

Cores CHNYT06 and CHNYY09 were aged in different vehicles with Oil 34 and Oil 33 respectively. Ford made these cores available to SwRI for PIXE and surface area tests. Core CHNYT09 was aged on a vehicle with engine Oil 33. The core had about 2.7 times the weight percent of phosphorus compared to Core 13 which was aged with Oil 33 in the FOCAS™ Rig. The PIXE results for Core CHNYT06 indicated no 0.0 weight percent phosphorus. This is the same phosphorus result recorded for Core 3. Oil 34 had no phosphorus.

### C. Surface Area Results

SwRI completed the surface area testing on the four aged cores and the two samples from Ford. The surface area results are given in Table 2.

**Table 2 – Surface Area Results**

Catalyst	Surface Area Results
Ford CHNYT09-33	3.52 +/- 0.15 m <sup>2</sup> /g
Ford CHNYT06-34	8.86 +/- 0.86 m <sup>2</sup> /g
Core 13 - Oil 33	24.44 +/- 0.61 m <sup>2</sup> /g
Core 3 - Oil 34	24.69 +/- 0.53 m <sup>2</sup> /g
Core 25 - Oil A	25.20 +/- 0.63 m <sup>2</sup> /g
Core 20 - Oil B	25.99 +/- 1.25 m <sup>2</sup> /g

## III. CONCLUSIONS

SwRI has completed its obligations for the OPEST II Program using Oils A, B, 33, and 34. The conclusions for the action items discussed in this letter are as follows:

### A. SGR Conversion Efficiency Temperature Study

The OPEST II data from the enhanced SGR procedure were used to calculate conversion efficiencies at temperatures other than 350°C. It appears that either 290°C or 300°C is more appropriate for a conversion efficiency temperature than 350°F with the enhanced SGR procedure. Conversion efficiencies calculated at 290°C or 300°C discriminate Oil A and B as well as Oil 33 and 34 for all three emission constituents (HC, CO, and NO). In comparing the average HC, CO and NO<sub>x</sub> efficiencies between the appropriate oils, efficiencies calculated at 290°C and 300°C produced similar results. Thus, one is not more "optimal" than another. The conversion temperature of 290°C was selected for future testing.

### B. Proton-Induced X-Ray Emissions

For the catalysts tested in the OPEST II program, the amount of phosphorus in the fresh oil does not correlate directly with the percent phosphorus in the catalyst core sample as determined by the PIXE method. The PIXE result for phosphorus in core 13 (2.397 wt%) was greater than in core 25 (2.089 wt%) even though the phosphorus content in fresh Oil 33 (0.10%) was less than in Oil A (0.11%). Refer to the graph in Appendix B.

The Ford catalyst formulation is different from the OPEST II catalyst formulation and the Ford catalysts were aged in vehicles rather than the FOCAS™ Rig. Therefore, the absolute value of the results are different but the catalysts do rank Oil 33 and Oil 34 in the same order as the OPEST II procedure for the amount of phosphorus, zinc, and calcium in the sample.

### C. Surface Area Results

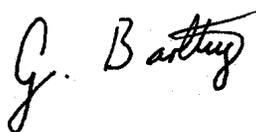
Within the error limits, all of the OPEST II cores have the same aged surface area. The Ford catalyst formulation is different from the OPEST II catalyst formulation. The catalysts supplied by Ford were each aged on a vehicle in a relatively hot location, their lower surface areas are probably a result of thermal degradation, and fairly typical of vehicle aged converters.

If you have any questions and/or comments, please send them to Brent Shoffner at SwRI on (210) 522-6986 or via e-mail [bshoffner@swri.org](mailto:bshoffner@swri.org).



---

Cynthia Webb



---

Gordon Bartley



---

Brent Shoffner

**APPENDIX A – OPEST II 200-Hour Aged Catalyst Efficiency Comparison at  
Various Temperatures**

OPEST II 200-Hr Aged Catalyst Conversion Efficiency  
Comparisons at Various Temperatures

I. Data and Definitions

Four catalysts were aged for 200 hrs. One oil was run in each of the catalysts. Four light-off tests were run at 120° start temperature in each catalyst. Stoichiometric perturbed light-offs at  $T_{50}$  and efficiencies at various temperatures were measured for each of the four tests run on each catalyst. Efficiencies were computed at 250°, 260°, 270°, 280°, 290°, and 300°C. The following table lists the oils run on each catalyst.

Table 1. Catalyst and Oil Matchups

Catalyst	Oil
3	34
13	33
20	B
25	A

II. 200-Hr Aged Catalyst Conversion Efficiency Data

The two questions to be answered for these data include:

- (1) Are the average efficiencies at Oil A different than at Oil B?
- (2) Are the average efficiencies at Oil 33 different than at Oil 34?

- a. Efficiency at 250°C

Upon examination of the variability across the catalysts, it was determined that there was not a statistically significant difference in the standard deviations across the four catalysts with respect to the HC and CO efficiency. Thus, an analysis of variance (ANOVA) test was used to compare the average efficiencies among the oils. However, there was a significant difference in the standard deviations across the four catalysts for the NO<sub>x</sub> efficiency. In this case, a nonparametric Kruskal-Wallis test was used to compare the medians of the NO<sub>x</sub> efficiency. Table 2 lists the conclusions in comparing the efficiencies of the two oil groups. All statistical tests were made at the 5% level of significance (i.e., p-values < 0.05 indicate significant differences in the averages or the medians). Figures 1 and 2 illustrate the 95% intervals about the mean for HC and CO, respectively. Figure 3 illustrates the scatterplot of the NO<sub>x</sub> efficiency by oils.

Table 2. Conversion Efficiency Test Comparisons at 250°C

Response	Analysis Procedure	p-value	Oil A Compared to Oil B	Oil 33 Compared to Oil 34
HC Efficiency	ANOVA	0.0003	Oil A = Oil B	Oil 33 < Oil 34
CO Efficiency	ANOVA	0.0001	Oil A = Oil B	Oil 33 < Oil 34
NO <sub>x</sub> Efficiency	Kruskal-Wallis	0.0091	Oil A < Oil B	Oil 33 = Oil 34

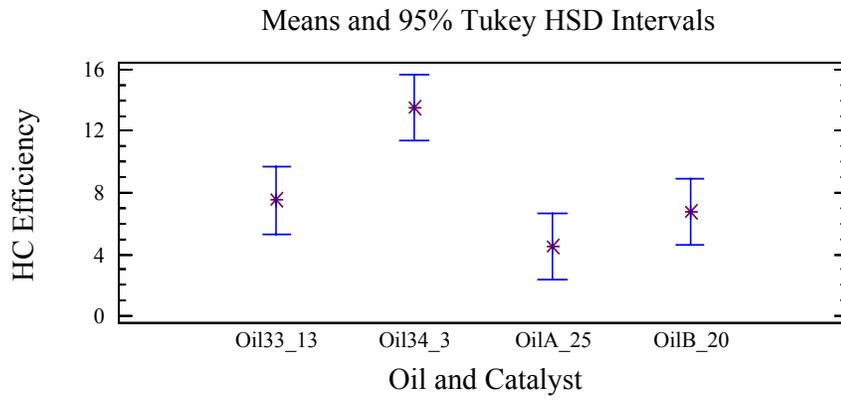


Figure 1. Interval Plot for HC Efficiency at 250°C

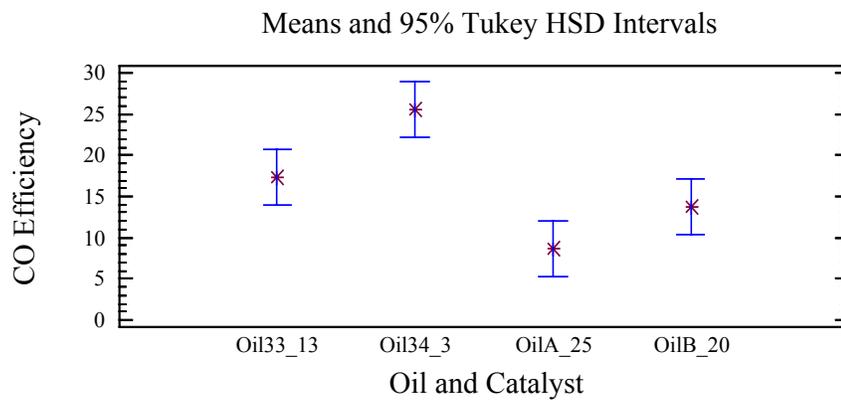


Figure 2. Interval Plot for CO Efficiency at 250°C

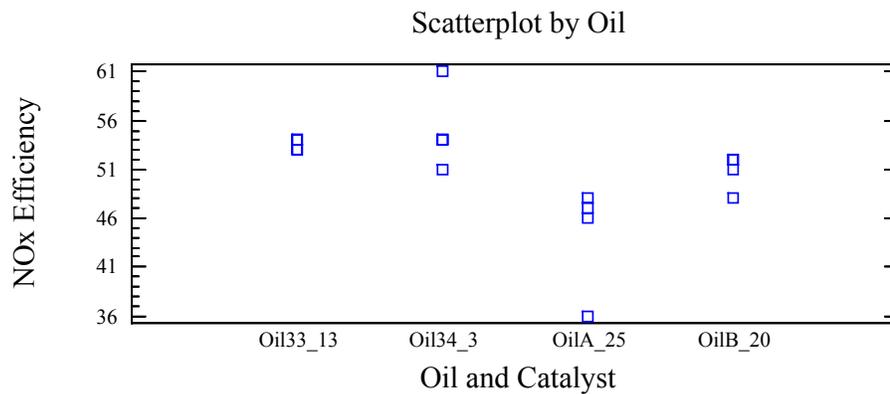


Figure 3. Scatterplot for NOx Efficiency at 250°C

b. Efficiency at 260°C

Upon examination of the variability across the catalysts, it was determined that there was not a statistically significant difference in the standard deviations across the four catalysts with respect to the HC, CO, and NOx efficiency. Thus, an analysis of variance (ANOVA) test was used to compare the average efficiencies among the oils. Table 3 lists the conclusions in comparing the average efficiency of the two oil groups. All statistical tests were made at the 5% level of significance (i.e., p-values < 0.05 indicate significant differences in the averages). Figures 4-6 illustrate the intervals about the mean for HC, CO and NOx, respectively.

Table 3. Conversion Efficiency Test Comparisons at 260°C

Response	Analysis Procedure	p-value	Oil A Compared to Oil B	Oil 33 Compared to Oil 34
HC Efficiency	ANOVA	0.0001	Oil A = Oil B	Oil 33 < Oil 34
CO Efficiency	ANOVA	0.0020	Oil A = Oil B	Oil 33 = Oil 34
NOx Efficiency	ANOVA	0.0054	Oil A = Oil B	Oil 33 = Oil 34

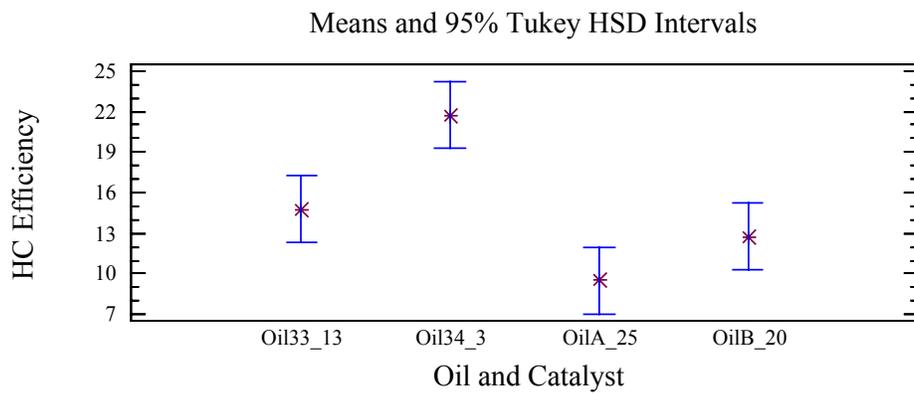


Figure 4. Interval Plot for HC Efficiency at 260°C

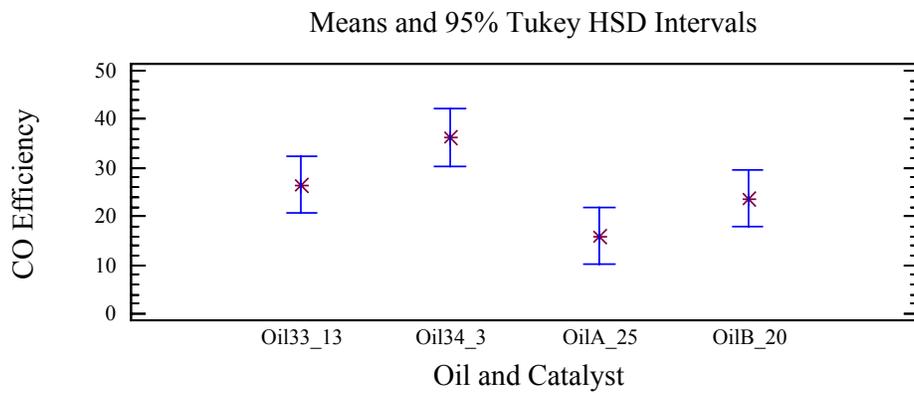


Figure 5. Interval Plot for CO Efficiency at 260°C

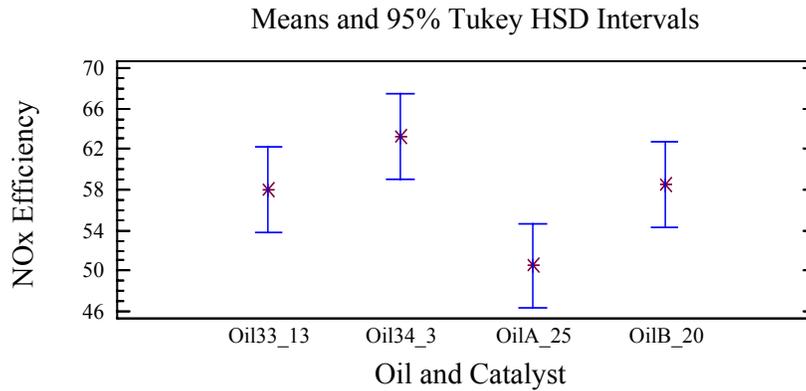


Figure 6. Interval Plot for NOx Efficiency at 260°C

c. Efficiency at 270°C

Upon examination of the variability across the catalysts, it was determined that there was not a statistically significant difference in the standard deviations across the four catalysts with respect to the HC and NOx efficiency. Thus, an analysis of variance (ANOVA) test was used to compare the average efficiencies among the oils. However, there was a significant difference in the standard deviations across the four catalysts for the CO efficiency. In this case, a nonparametric Kruskal-Wallis test was used to compare the medians of the CO efficiency. Table 4 lists the conclusions in comparing the efficiencies of the two oil groups. All statistical tests were made at the 5% level of significance (i.e., p-values < 0.05 indicate significant differences in the averages or the medians). Figures 7 and 9 illustrate the intervals about the mean for HC and NOx, respectively. Figure 8 illustrates the scatterplot of the CO efficiency by oils.

Table 4. Conversion Efficiency Test Comparisons at 270°C

Response	Analysis Procedure	p-value	Oil A Compared to Oil B	Oil 33 Compared to Oil 34
HC Efficiency	ANOVA	0.0003	Oil A = Oil B	Oil 33 < Oil 34
CO Efficiency	Kruskal-Wallis	0.0155	Oil A = Oil B	Oil 33 < Oil 34
NOx Efficiency	ANOVA	0.0077	Oil A < Oil B	Oil 33 = Oil 34

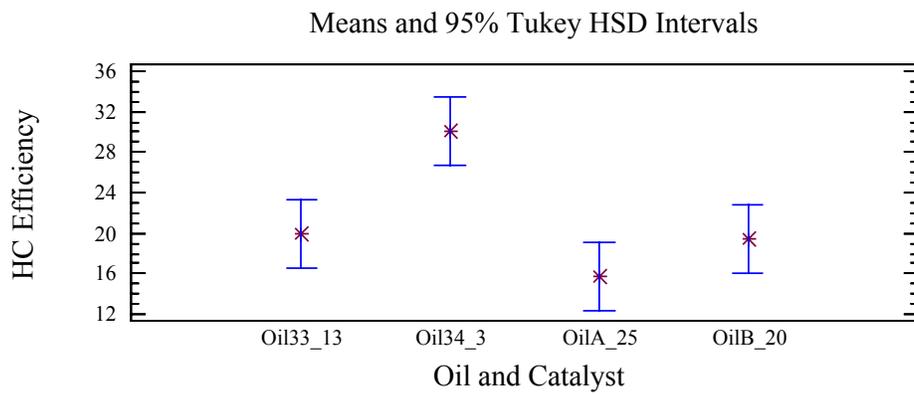


Figure 7. Interval Plot for HC Efficiency at 270°C

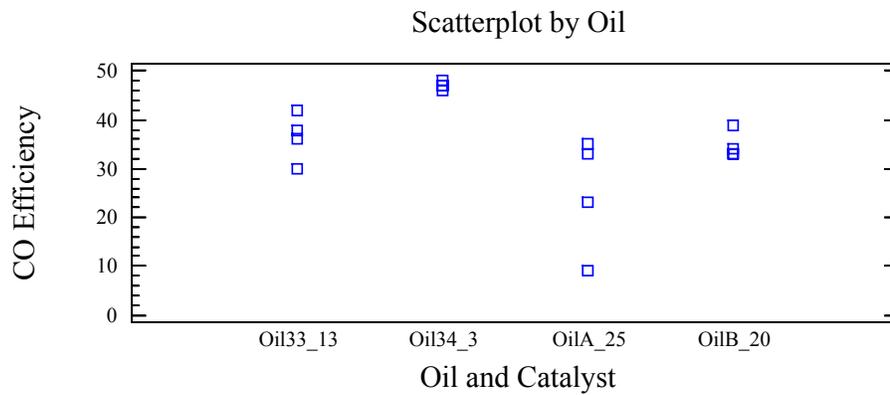


Figure 8. Scatterplot for CO Efficiency at 270°C

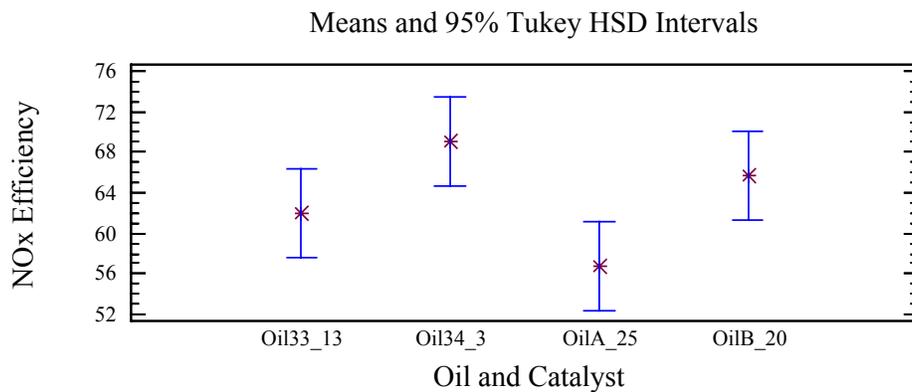


Figure 9. Interval Plot for NOx Efficiency at 270°C

d. Efficiency at 280°C

Upon examination of the variability across the catalysts, it was determined that there was not a statistically significant difference in the standard deviations across the four catalysts with respect to the HC and NOx efficiency. Thus, an analysis of variance (ANOVA) test was used to compare the average efficiencies among the oils. However, there was a significant difference in the standard deviations across the four catalysts for the CO efficiency. In this case, a nonparametric Kruskal-Wallis test was used to compare the medians of the CO and NOx efficiencies. Table 5 lists the conclusions in comparing the efficiency of the two oil groups. All statistical tests were made at the 5% level of significance (i.e., p-values < 0.05 indicate significant differences in the averages or the medians). Figures 10 and 12 illustrate the intervals about the mean for HC and NOx, respectively. Figure 11 illustrates the scatterplot of the CO efficiency by oils.

Table 5. Conversion Efficiency Test Comparisons at 280°C

Response	Analysis Procedure	p-value	Oil A Compared to Oil B	Oil 33 Compared to Oil 34
HC Efficiency	ANOVA	0.0001	Oil A < Oil B	Oil 33 < Oil 34
CO Efficiency	Kruskal-Wallis	0.0120	Oil A = Oil B	Oil 33 = Oil 34
NOx Efficiency	ANOVA	0.0046	Oil A < Oil B	Oil 33 < Oil 34

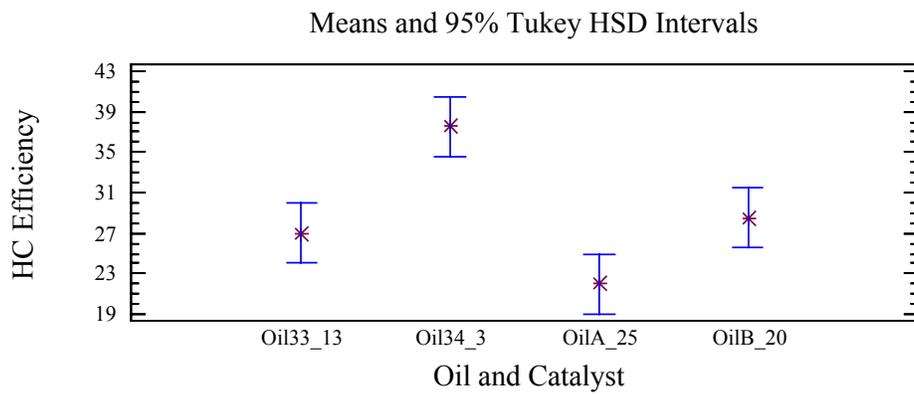


Figure 10. Interval Plot for HC Efficiency at 280°C

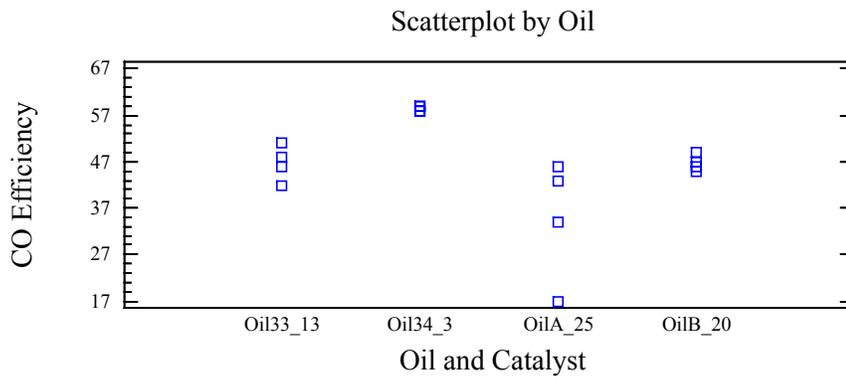


Figure 11. Scatterplot for CO Efficiency at 280°C

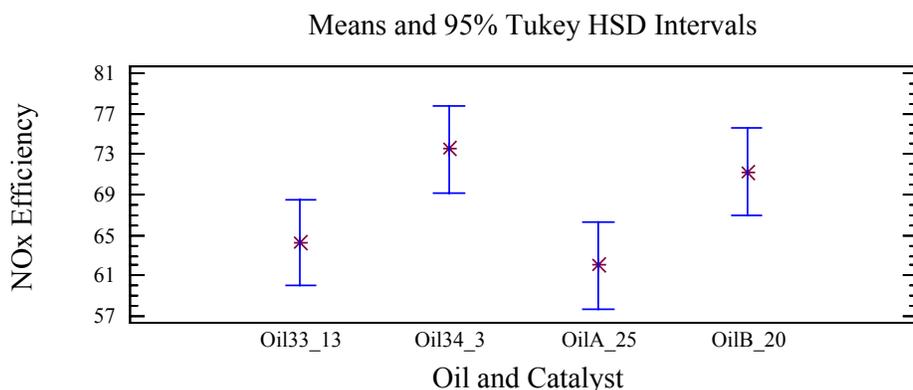


Figure 12. Interval Plot for NOx Efficiency at 280°C

e. Efficiency at 290°C

Upon examination of the variability across the catalysts, it was determined that there was not a statistically significant difference in the standard deviations across the four catalysts with respect to the HC and NOx efficiency. Thus, an analysis of variance (ANOVA) test was used to compare the average efficiency among the oils. However, there was a significant difference in the standard deviations across the four catalysts for the CO efficiency. In this case, a nonparametric Kruskal-Wallis test was used to compare the medians of the CO efficiency. Table 6 lists the conclusions in comparing the efficiencies of the two oil groups. All statistical tests were made at the 5% level of significance (i.e., p-values < 0.05 indicate significant differences in the averages or the medians). Figures 13 and 15 illustrate the intervals about the mean for HC and NOx, respectively. Figure 14 illustrates the scatterplot of the CO efficiency by oils.

Table 6. Conversion Efficiency Test Comparisons at 290°C

Response	Analysis Procedure	p-value	Oil A Compared to Oil B	Oil 33 Compared to Oil 34
HC Efficiency	ANOVA	0.0001	Oil A < Oil B	Oil 33 < Oil 34
CO Efficiency	Kruskal-Wallis	0.0068	Oil A < Oil B	Oil 33 < Oil 34
NOx Efficiency	ANOVA	0.0005	Oil A < Oil B	Oil 33 < Oil 34

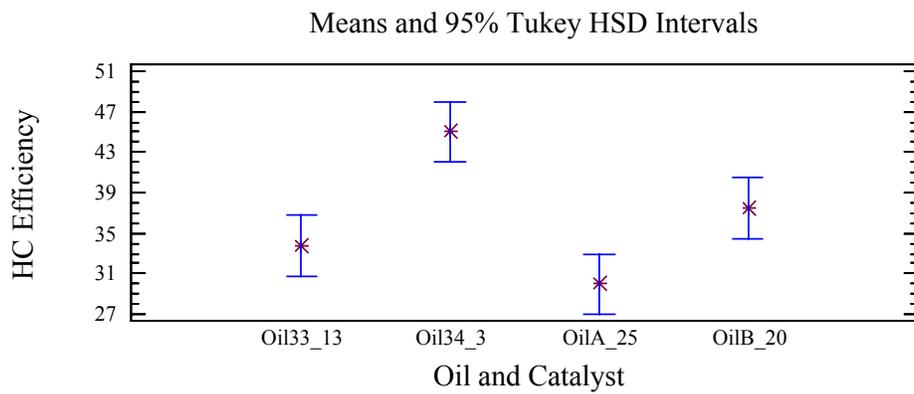


Figure 13. Interval Plot for HC Efficiency at 290°C

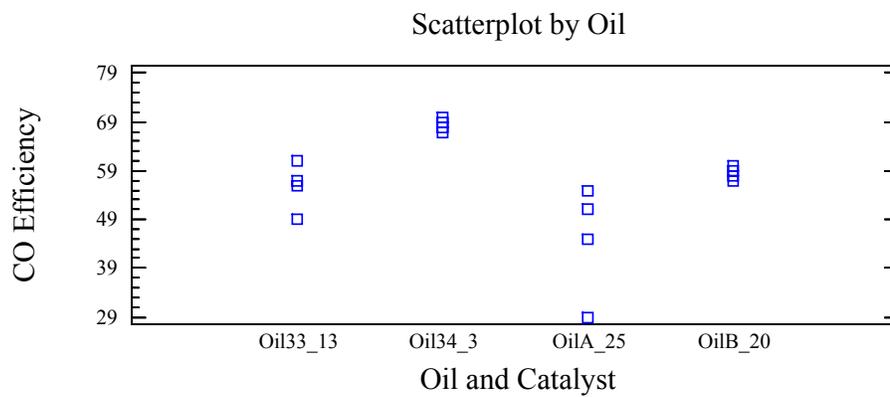


Figure 14. Scatterplot for CO Efficiency at 290°C

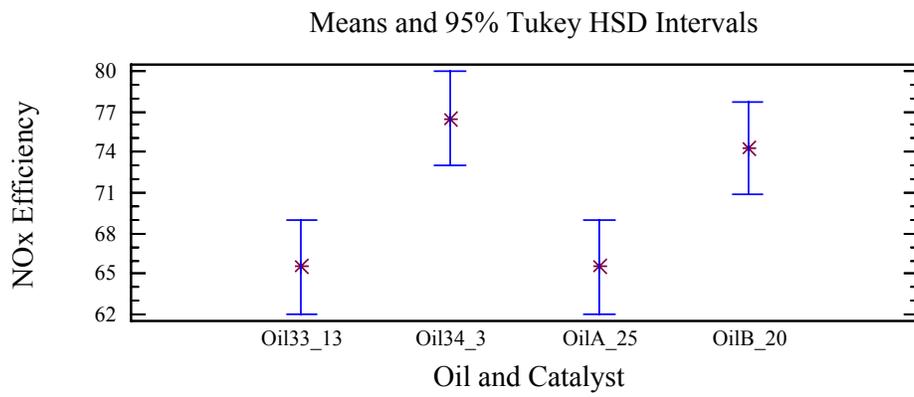


Figure 15. Interval Plot for NOx Efficiency at 290°C

f. Efficiency at 300°C

Upon examination of the variability across the catalysts, it was determined that there was not a statistically significant difference in the standard deviations across the four catalysts with respect to the HC and NOx efficiency. Thus, an analysis of variance (ANOVA) test was used to compare the average efficiency among the oils. However, there was a significant difference in the standard deviations across the four catalysts for the CO efficiency. In this case, a nonparametric Kruskal-Wallis test was used to compare the medians of the CO efficiencies. Table 7 lists the conclusions in comparing the efficiencies of the two oil groups. All statistical tests were made at the 5% level of significance (i.e., p-values < 0.05 indicate significant differences in the averages or the medians). Figures 16 and 18 illustrate the intervals about the mean for HC and NOx, respectively. Figure 17 illustrates the scatterplot of the CO efficiency by oils.

Table 7. Conversion Efficiency Test Comparisons at 300°C

Response	Analysis Procedure	p-value	Oil A Compared to Oil B	Oil 33 Compared to Oil 34
HC Efficiency	ANOVA	0.0001	Oil A < Oil B	Oil 33 < Oil 34
CO Efficiency	Kruskal-Wallis	0.0056	Oil A < Oil B	Oil 33 < Oil 34
NOx Efficiency	ANOVA	0.0001	Oil A < Oil B	Oil 33 < Oil 34

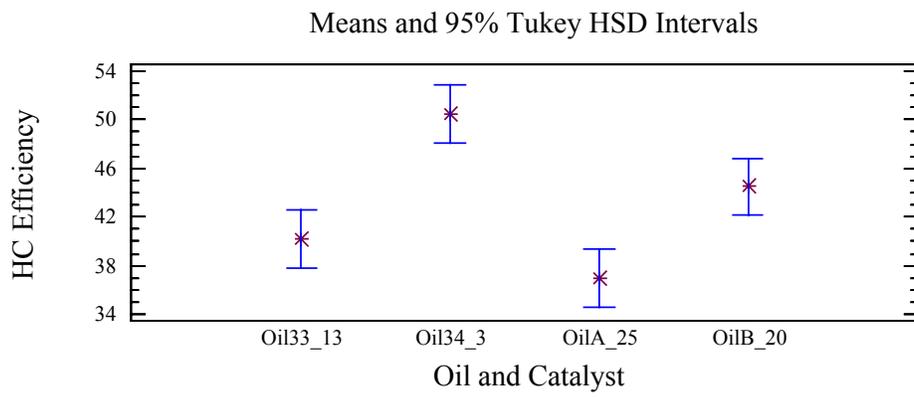


Figure 16. Interval Plot for HC Efficiency at 300°C

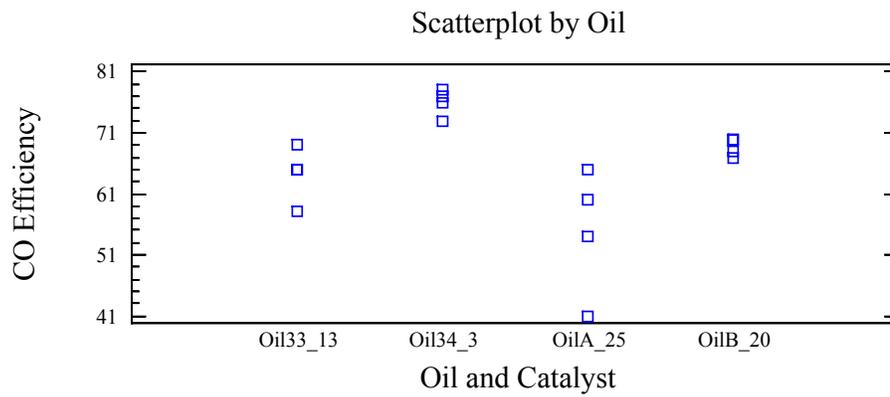


Figure 17. Scatterplot for CO Efficiency at 300°C

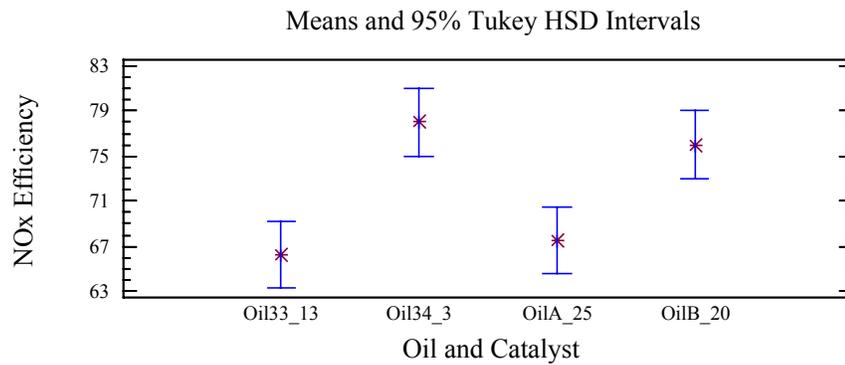
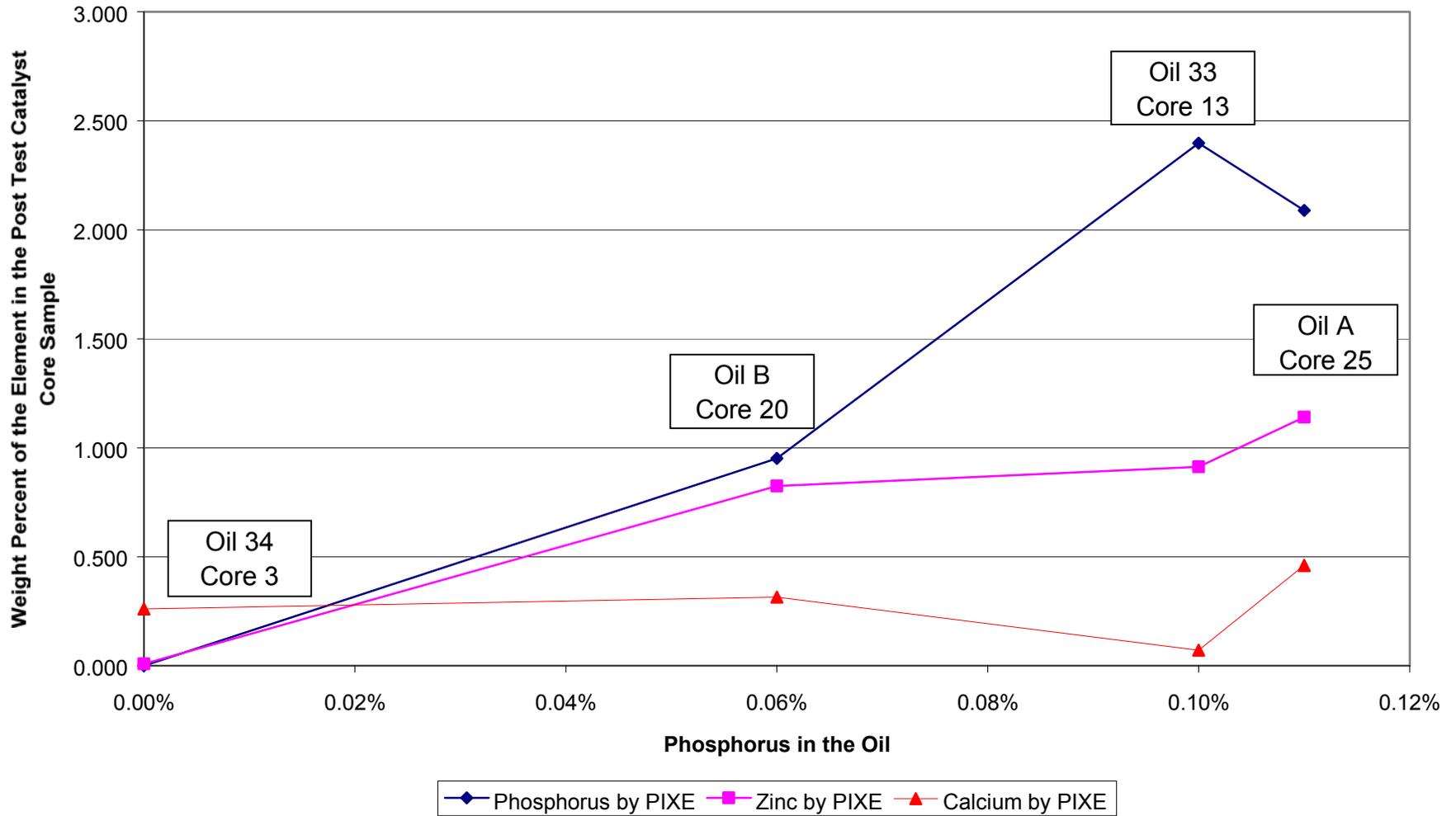


Figure 18. Interval Plot for NOx Efficiency at 300°C

## **APPENDIX B – Proton-Induced X-Ray Emissions Results**

**OPEST II - Proton-Induced X-Ray Emission (PIXE) Results  
From the Post Test Catalyst Core Sample Versus  
Percent Phosphorus in the Test Oil**



**APPENDIX C – Stoichiometric Perturbated  
Catalyst Core Efficiencies @ 290°C**

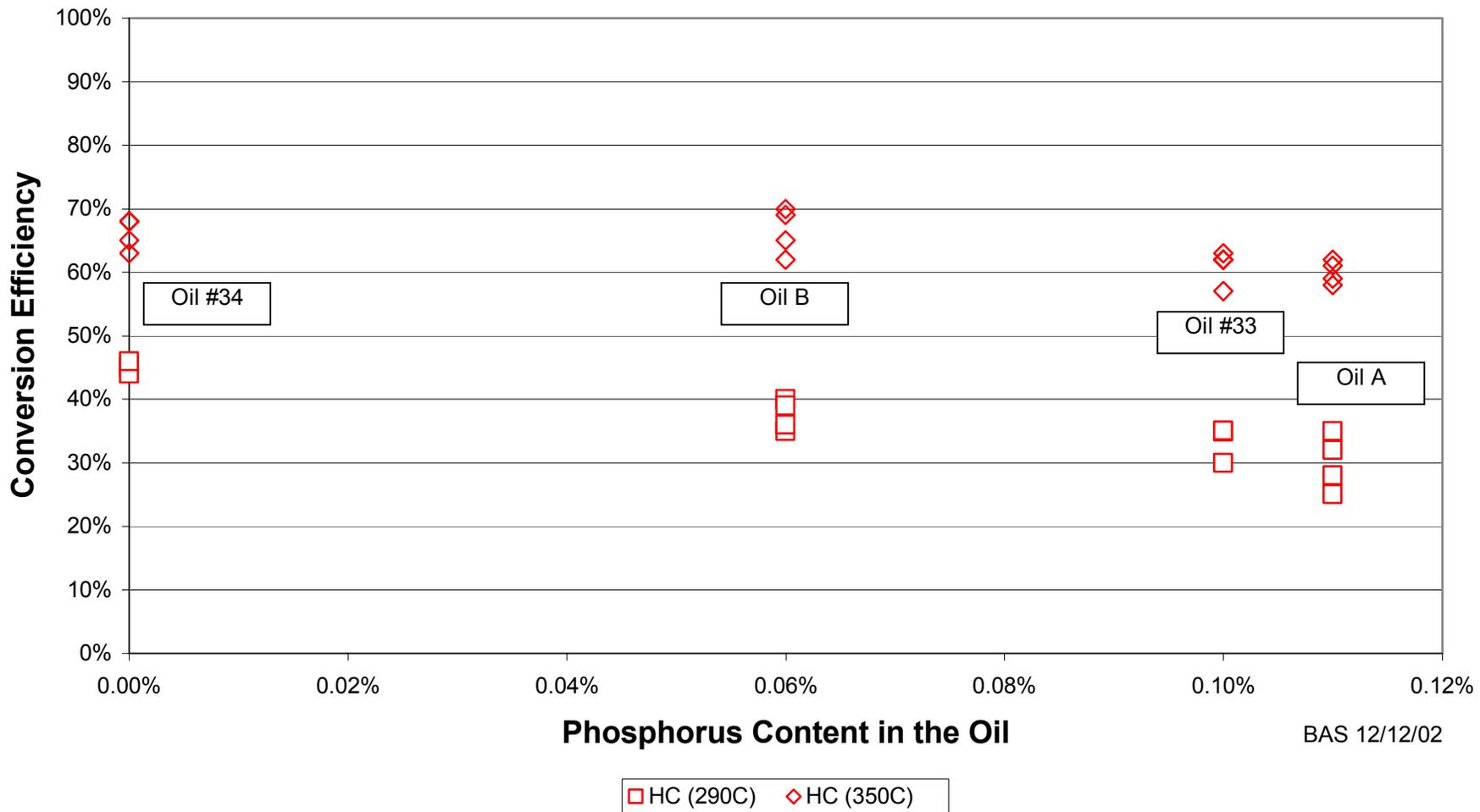
### Catalyst Cores Used in the OPEST II Program

Catalyst I.D. Number	Test Date	Stoichiometric Perturbated Efficiencies @ 290°C, percent		
		HC	CO	NOx
3 run 1	04/29/02	45	67	79
3 run 2	04/29/02	45	69	74
3 run 3	04/30/02	44	68	76
3 run 4	04/30/02	46	70	77
20 run 1	04/29/02	40	59	79
20 run 2	04/29/02	39	60	69
20 run 3	04/30/02	35	58	76
20 run 4	04/30/02	36	57	73
25 run 1	04/29/02	35	55	64
25 run 2	04/29/02	32	51	66
25 run 3	04/30/02	25	29	62
25 run 4	04/30/02	28	45	70
13 run 1	04/29/02	35	57	65
13 run 2	04/29/02	35	61	62
13 run 3	04/30/02	35	56	66
13 run 4	04/30/02	30	49	69

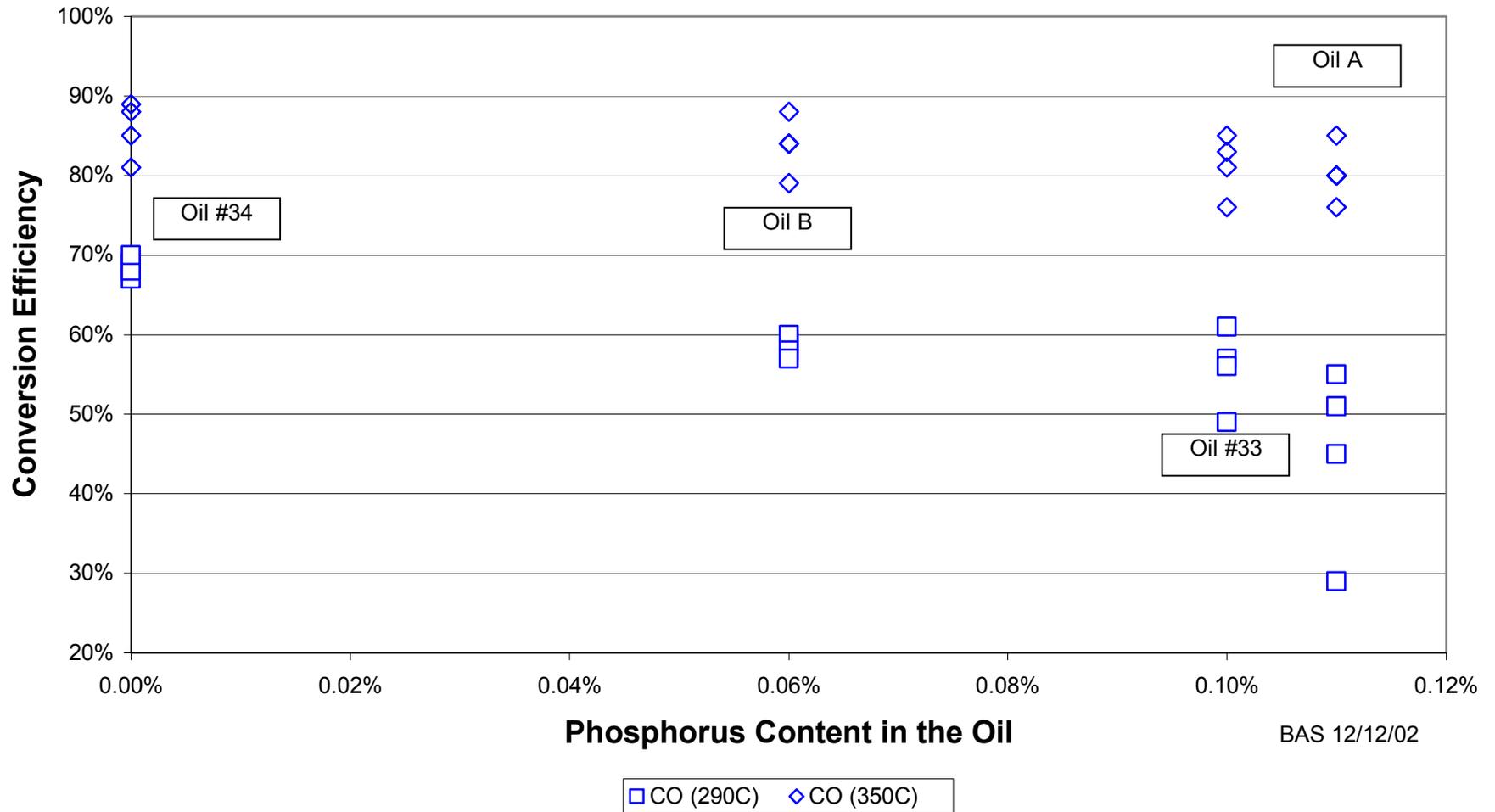
**OPEST II - Results - 200 Hours of Aging in the FOCAS(TM) Rig  
Synthetic Gas Reactor - *Enhanced* Stoichiometric Perturbated Procedure  
120 C Start Temperature**

Run	Date	Operational Validity	Oil Code	Oil Description	P	Core	Conv.@350C			Conv.@290C		
							HC	CO	NO	HC	CO	NO
1	4/29/02	Valid	#34	0.0% P w/GF-2 Ca/Mg detergent	0.00%	3	65%	81%	79%	45%	67%	79%
2	4/29/02	Valid	#34	0.0% P w/GF-2 Ca/Mg detergent	0.00%	3	68%	89%	79%	45%	69%	74%
3	4/30/02	Valid	#34	0.0% P w/GF-2 Ca/Mg detergent	0.00%	3	68%	88%	81%	44%	68%	76%
4	4/30/02	Valid	#34	0.0% P w/GF-2 Ca/Mg detergent	0.00%	3	63%	85%	80%	46%	70%	77%
1	4/29/02	Valid	B reblend	0.06% P - fully formulated	0.06%	20	70%	84%	80%	40%	59%	79%
2	4/29/02	Valid	B reblend	0.06% P - fully formulated	0.06%	20	69%	88%	77%	39%	60%	69%
3	4/30/02	Valid	B reblend	0.06% P - fully formulated	0.06%	20	62%	79%	81%	35%	58%	76%
4	4/30/02	Valid	B reblend	0.06% P - fully formulated	0.06%	20	65%	84%	78%	36%	57%	73%
1	4/29/02	Valid	A reblend	0.11% P - fully formulated	0.11%	25	62%	85%	69%	35%	55%	64%
2	4/29/02	Valid	A reblend	0.11% P - fully formulated	0.11%	25	58%	80%	73%	32%	51%	66%
3	4/30/02	Valid	A reblend	0.11% P - fully formulated	0.11%	25	61%	80%	73%	25%	29%	62%
4	4/30/02	Valid	A reblend	0.11% P - fully formulated	0.11%	25	59%	76%	74%	28%	45%	70%
1	4/29/02	Valid	#33	0.10% P with no detergent	0.10%	13	62%	81%	70%	35%	57%	65%
2	4/29/02	Valid	#33	0.10% P with no detergent	0.10%	13	62%	85%	68%	35%	61%	62%
3	4/30/02	Valid	#33	0.10% P with no detergent	0.10%	13	63%	83%	72%	35%	56%	66%
4	4/30/02	Valid	#33	0.10% P with no detergent	0.10%	13	57%	76%	71%	30%	49%	69%

**OPEST II - HC - 200 Hrs. Aging**  
**Conversion Efficiency at 290C and 350C - Synthetic Gas Reactor**  
**Enhanced Stoichiometric Perturbated Procedure**

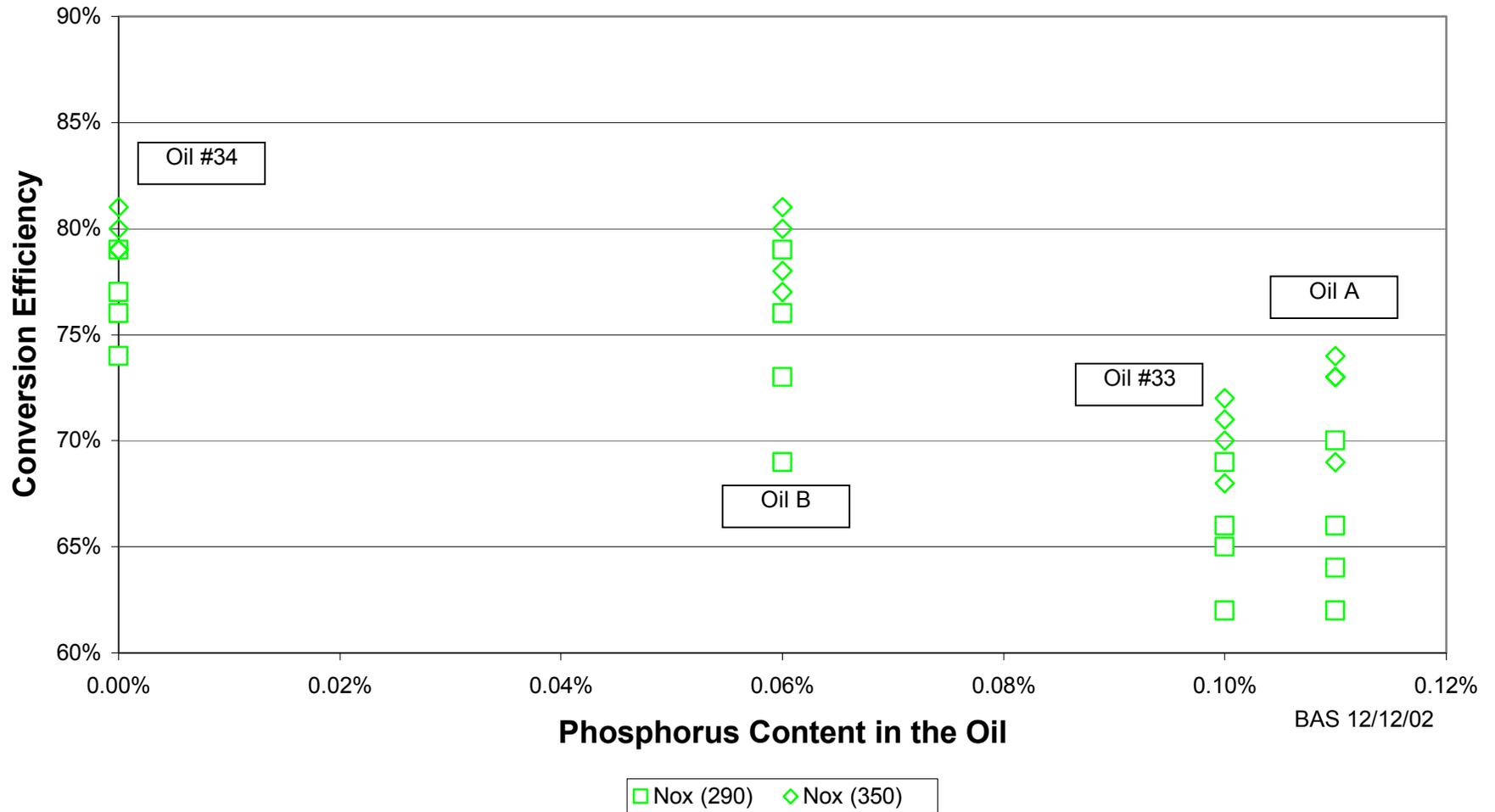


**OPEST II - CO - 200 Hrs. Aging**  
**Conversion Efficiency at 290C and 350C - Synthetic Gas Reactor**  
**Enhanced Stoichiometric Perturbated Procedure**



BAS 12/12/02

**OPEST II - NOx - 200 Hrs. Aging**  
**Conversion Efficiency at 290C and 350C - Synthetic Gas Reactor**  
**Enhanced Stoichiometric Perturbated Procedure**



BAS 12/12/02

# Attachment No. 6 (2 of 2)

---

Gordon Bartley  
Janet Buckingham  
Brent Shoffner  
Cynthia Webb

June 28, 2005



# OPEST II Program History

presented to the

## Emission Systems Compatibility Improvement Team

---

Gordon Bartley

Janet Buckingham

Brent Shoffner

Cynthia Webb

June 28, 2005



# Why is a Presentation of the OPEST II FOCAS(R) Project Relevant?

---

- ◆ “Those who disregard history are doomed to relive it.”
- ◆ Catalyst evaluation after engine aging
  - Synthetic gas reactor (SGR)
    - » Used for OPEST II
- ◆ FOCAS could be used in conjunction with an engine test to precondition (de-green) test catalysts
- ◆ Experience with the test materials
  - catalyst specification / fuel / oil



# Presentation Overview

---

- ◆ Background
- ◆ Oil, Fuel, Catalysts
- ◆ Program Steps
  - Catalyst preconditioning - No oil injection
  - 200-hours aging w/test oil (Cynthia Webb)
  - Synthetic Gas Reactor (SGR)
    - » Enhancements for precision (Gordon Bartley)
- ◆ Results
- ◆ Conclusion
- ◆ Update of FOCAS



# OPEST II Background

---

- ◆ Oil Protection of Emissions System Test (OPEST)
  - Driving force - lower vehicle emission standards
  - In “Needs statement” for GF-3 and GF-4
- ◆ ASTM/SwRI Contract - May 2001
  - Amendment No. 2
  - Partial funding of OPEST II Matrix
    - » SwRI covered the remaining costs
- ◆ Presentation made to OPEST II Task Force
  - May 2002



# Test Oil

---

- ◆ One test each on four test oils (2 field pairs)
  - Oil B - 0.06% P oil re-blend (fully formulated)
  - Oil A - 0.11% P oil re-blend (fully formulated)
  - Oil 34 - 0.0% P oil with GF-2 Ca/Mg detergent
  - Oil 33 - 0.10% P oil with no detergent



# Catalysts

---

- ◆ Supplied by the Delphi Corporation
- ◆ (20) “precision” catalysts
- ◆ Close-coupled fast light off application
- ◆ Palladium:rhodium/9:1/60
- ◆ 600 cells per square inch
- ◆ 3.5 mil wall thickness



# Fuel

---

- ◆ California Phase II
- ◆ One batch used for the program
- ◆ Analytical results
  - Sulfur - 29 PPM
  - Phosphorus - 0.0 g/gallon
  - Lead -  $<.001$  g/gallon



# Program Steps - Preconditioning

---

- ◆ Preconditioned (4) catalysts
  - Catalysts 3, 13, 20, 25
- ◆ FOCAS™ Rig
  - Mild-thermal cycle
  - 20 hours
  - No oil injection
- ◆ Removed 1 in. dia. core from the front 1 in.
- ◆ Synthetic Gas Reactor (SGR) tests
  - Stoichiometric perturbed procedure



# Program Steps - 200 Aging Hours

---

- ◆ Reassemble the catalysts
- ◆ FOCAS™ Rig aging with test oils
  - Mild-thermal cycle
  - 200 hours

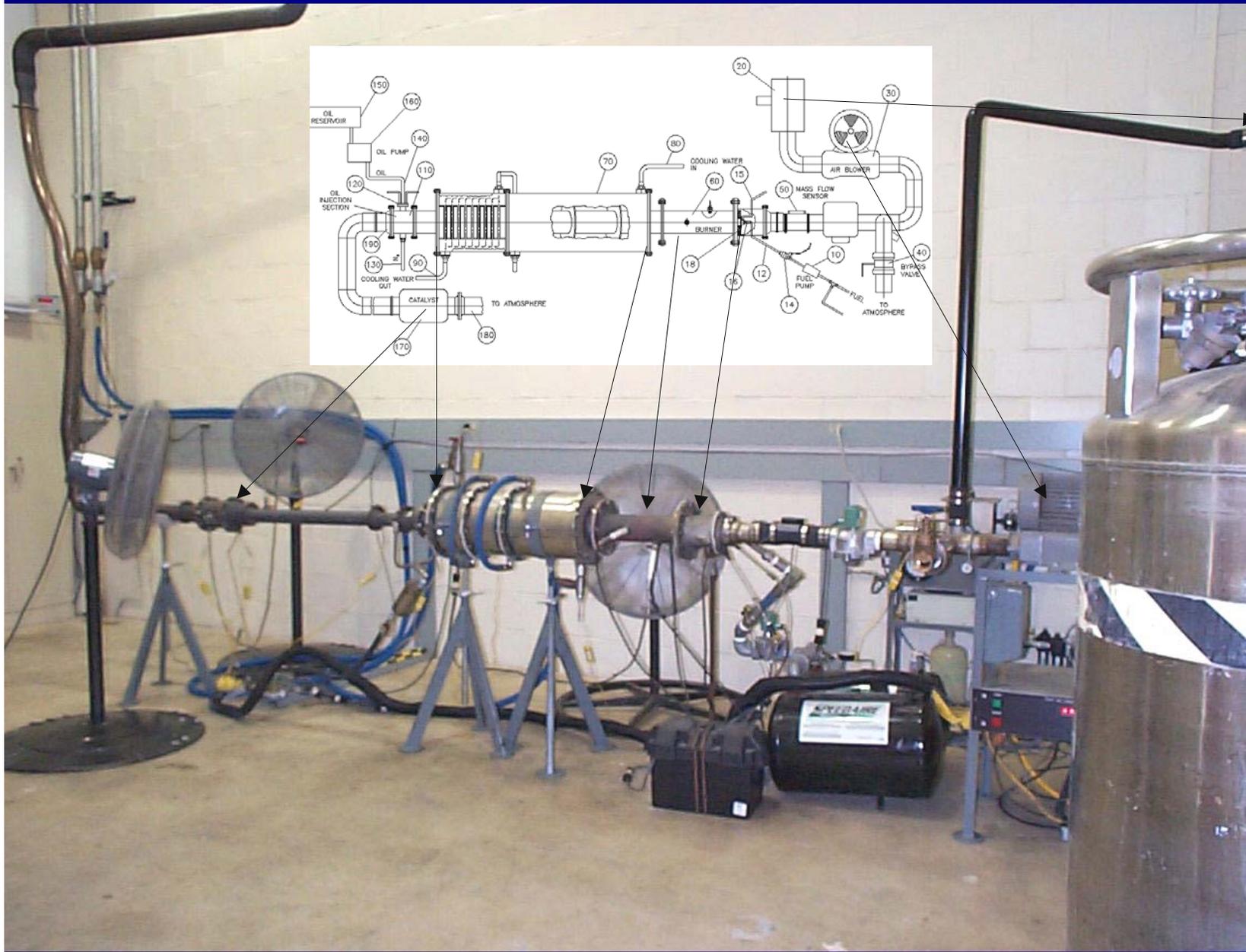


---

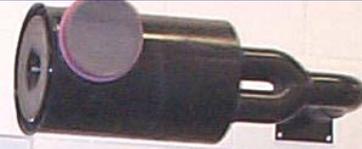
# FOCAS Overview



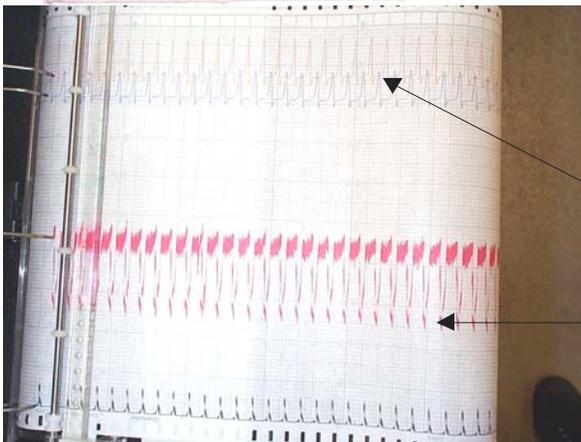
# FOCAST™ RIG IN CELL 20



FOCAS in cell 20



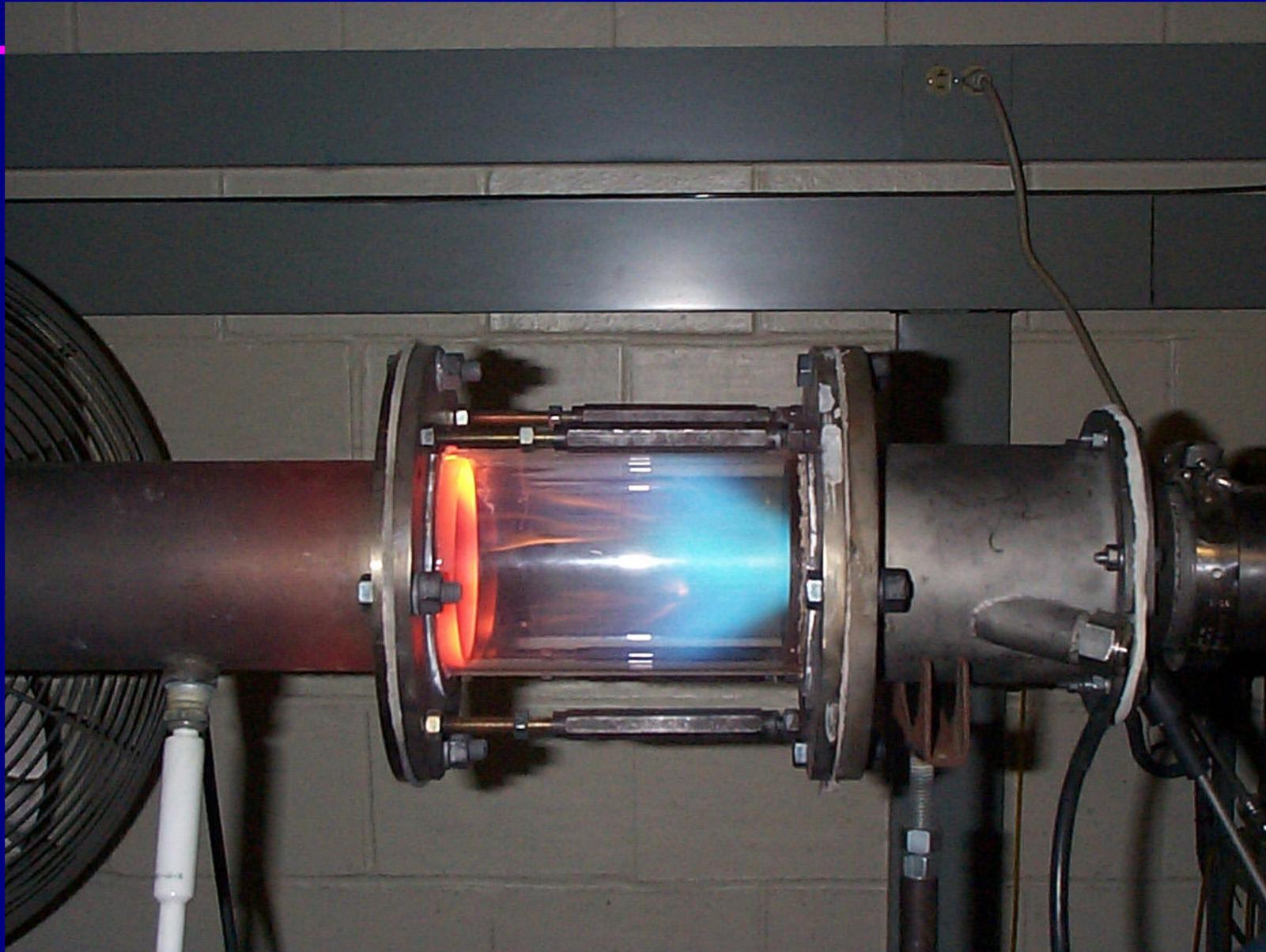
Control panel



AFR, Temperature output during RAT-A aging



# FOCAS™ BURNER WITH OPTIONAL QUARTZ WINDOW INSTALLED (USED FOR FUEL INJECTION WORK)



5/15/2002

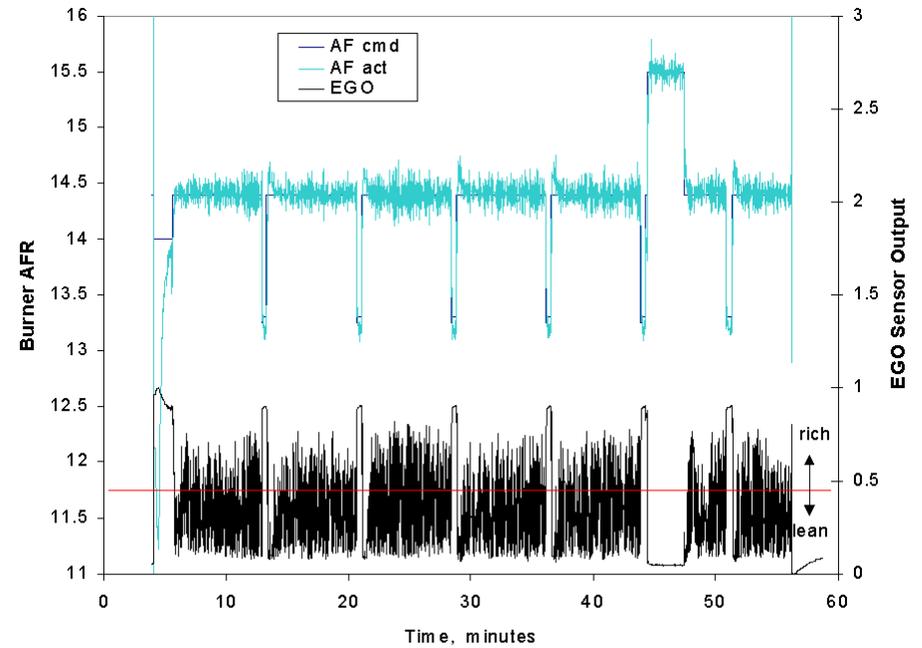
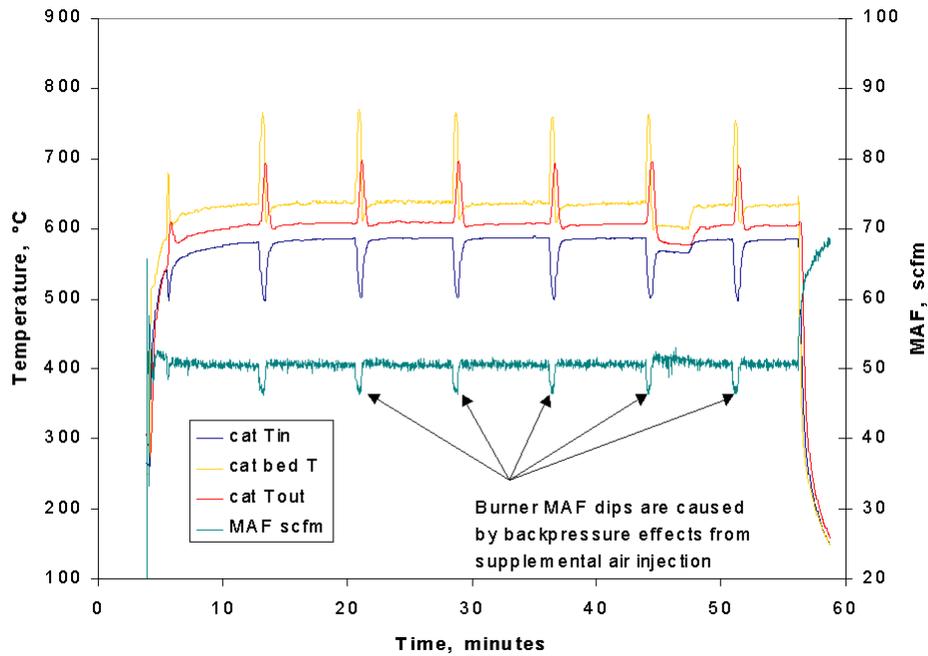
# Some Advantages:

---

- ◆ Low maintenance
- ◆ Minimal floor space
- ◆ Simplistic system
- ◆ Ease of cleaning between samples
- ◆ Precise, repeatable, durable
- ◆ Controllable oil consumption rate & character
- ◆ Capable of simulating many modes of operation



# MILD-THERMAL CYCLE



5/15/2002

# AGING ANALYSIS SOFTWARE

---

ANALYZES ALL MEASURED AGING  
DATA TO ASSESS PHYSICAL AGING  
CONDITIONS.

OUTPUTS:

AVERAGE AGING CONDITIONS  
TIME AT TEMPERATURE



5/15/2002

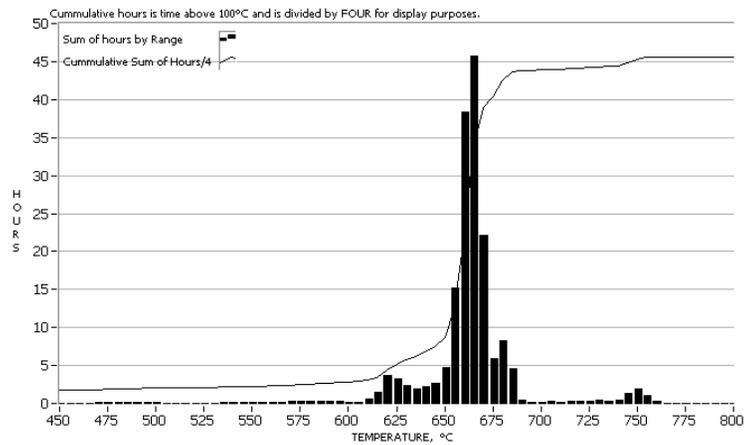
# Summary of Aging Analysis

AGING PARAMETER	AVERAGE AGING CONDITIONS				OPESTII Specification
	Catalyst Pair 1		Catalyst Pair 2		
	OILA	OILB	#33	#34	
AFR	14.30	14.29	14.41	14.28	na
EGO, volts	0.42	0.41	0.44	0.42	na
MAF, scfm	49.9	49.6	49.7	49.8	49 - 52
Catalyst Inlet T., 	594	592	582	587	580 - 640
Catalyst Bed T., 	660	653	640	652	na

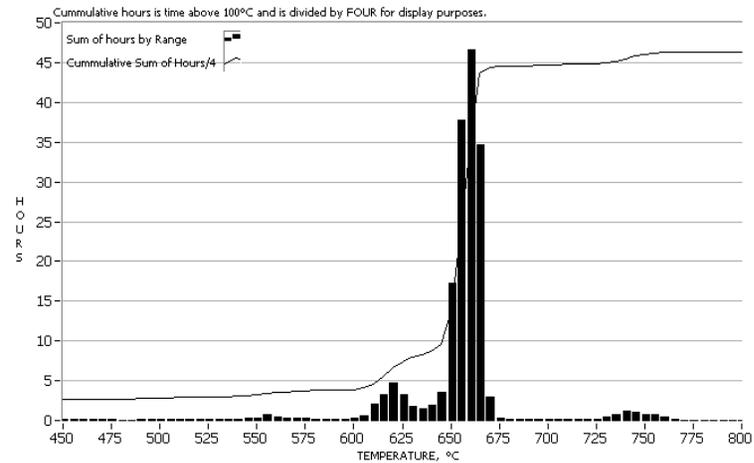


# Summary of Thermal Analysis

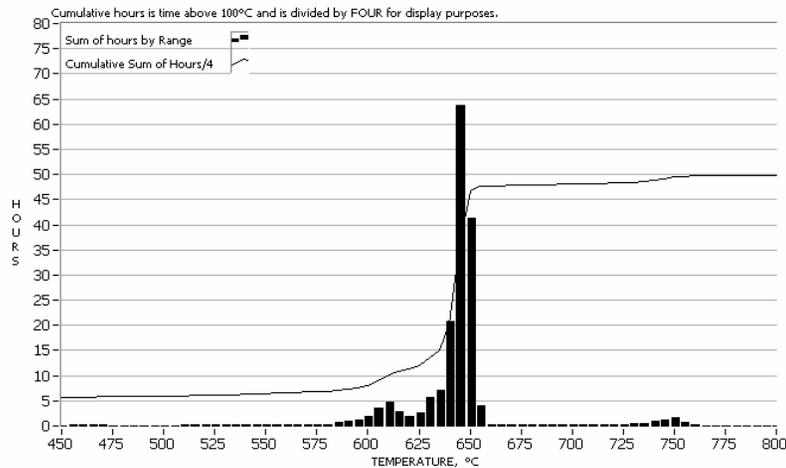
OPEST II OIL-A CATALYST  
MT CYCLE - 200 HRS  
CAT BED  
SUMMARY OF ALL MEASURED TEMPERATURE > 50°C



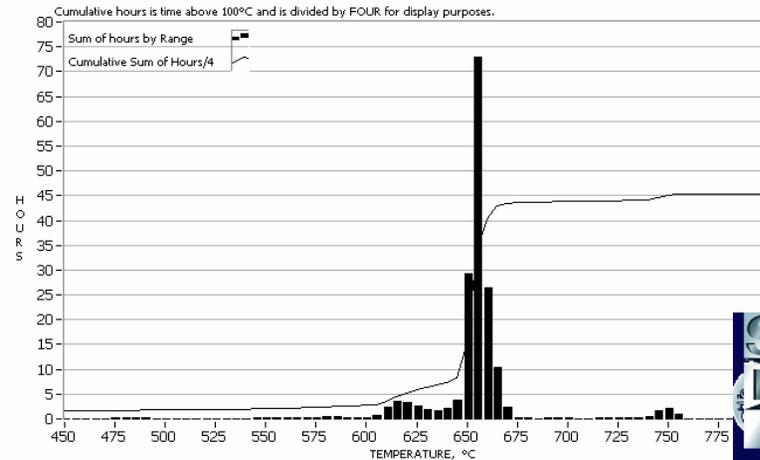
OPEST II OIL-B CATALYST  
MT CYCLE - 200 HRS  
CAT Bed  
SUMMARY OF ALL MEASURED TEMPERATURE > 50°C



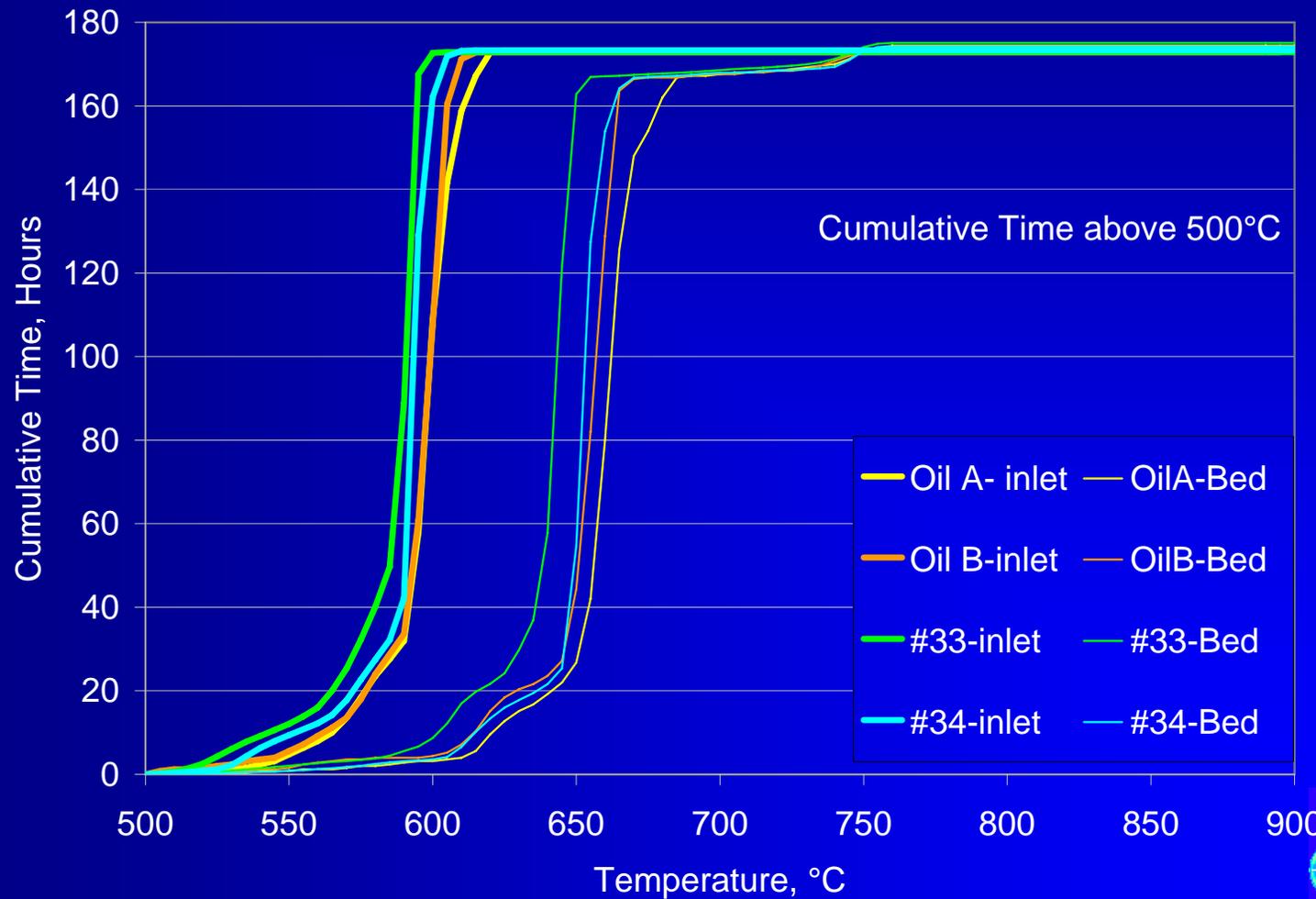
OPEST II OIL-#33 CATALYST  
MT CYCLE - 200 HRS  
CAT BED  
SUMMARY OF ALL MEASURED TEMPERATURE > 100°C



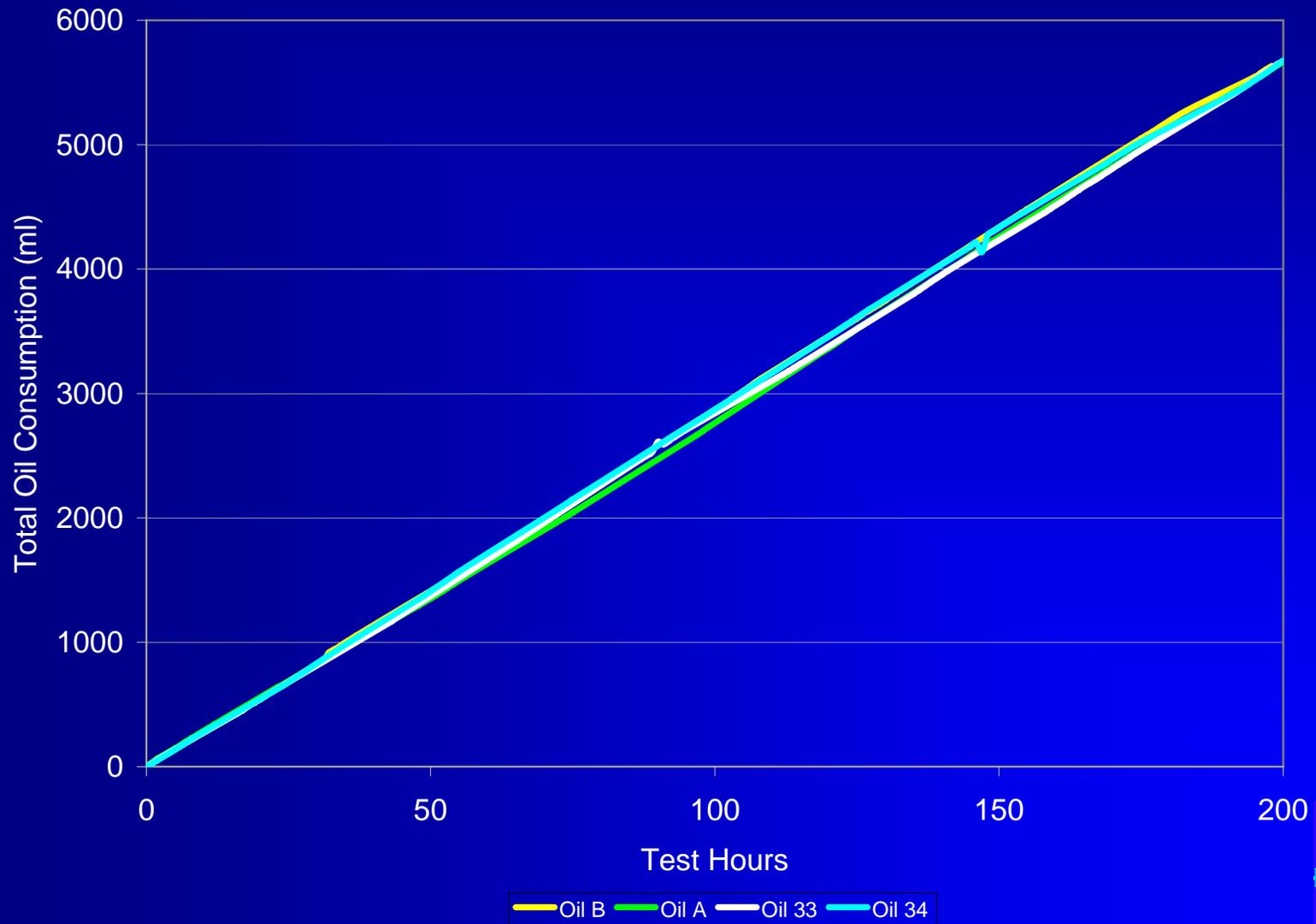
OPEST II OIL-#34 CATALYST  
MT CYCLE - 200 HRS  
CAT BED  
SUMMARY OF ALL MEASURED TEMPERATURE > 100°C



# Summary of Thermal Analysis

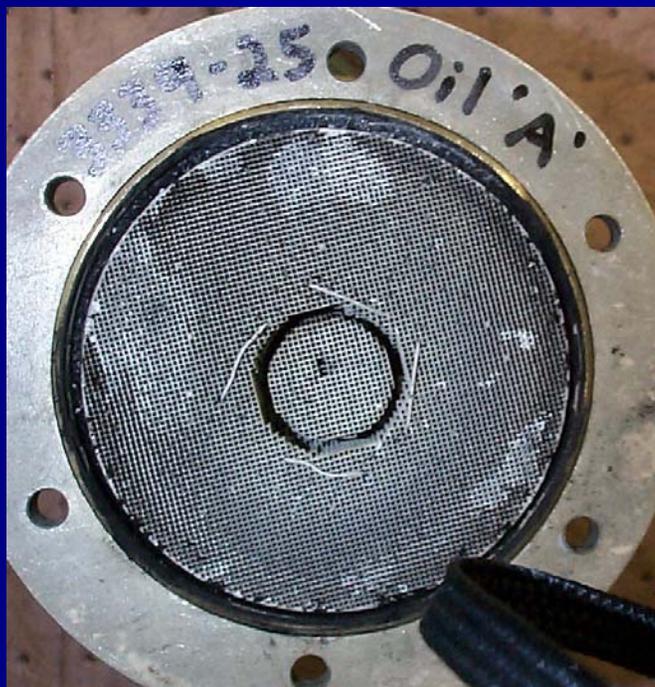


# OIL CONSUMPTION PROFILES



# Catalyst Pair: Oil A and Oil B

200 hours aging - OPEST MT-Cycle



## Oil A

Light, chalky. Brushes off when touched. Thin. Variations in photo from where catalyst face was touched



## Oil B

Hard Chalk deposits. Light gray. Deposits do not wipe off when touched.



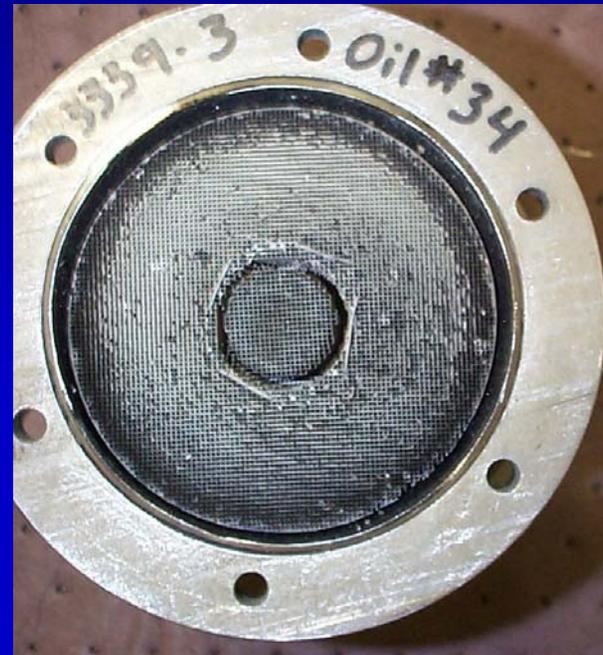
# Catalyst Pair: Oil #33 and Oil #34

200 hours aging - OPEST MT-Cycle



Oil #33

Thick, powdery deposit. Black, like soot. Physically blocking cells at outer edge. Wipes down into catalyst when touched.



Oil#34

Deposit building on face, looks like detergent? Light, chalky, but some stiffness. Deposits wipe down into catalyst and block cells when touched.



# Program Steps - SGR Enhancement

---

- ◆ The SGR rig was enhanced to improve
  - Precision
  - Equipment reproducibility
- ◆ Presentation by Gordon Bartley



# Synthetic Gas Reactor (SGR)

---

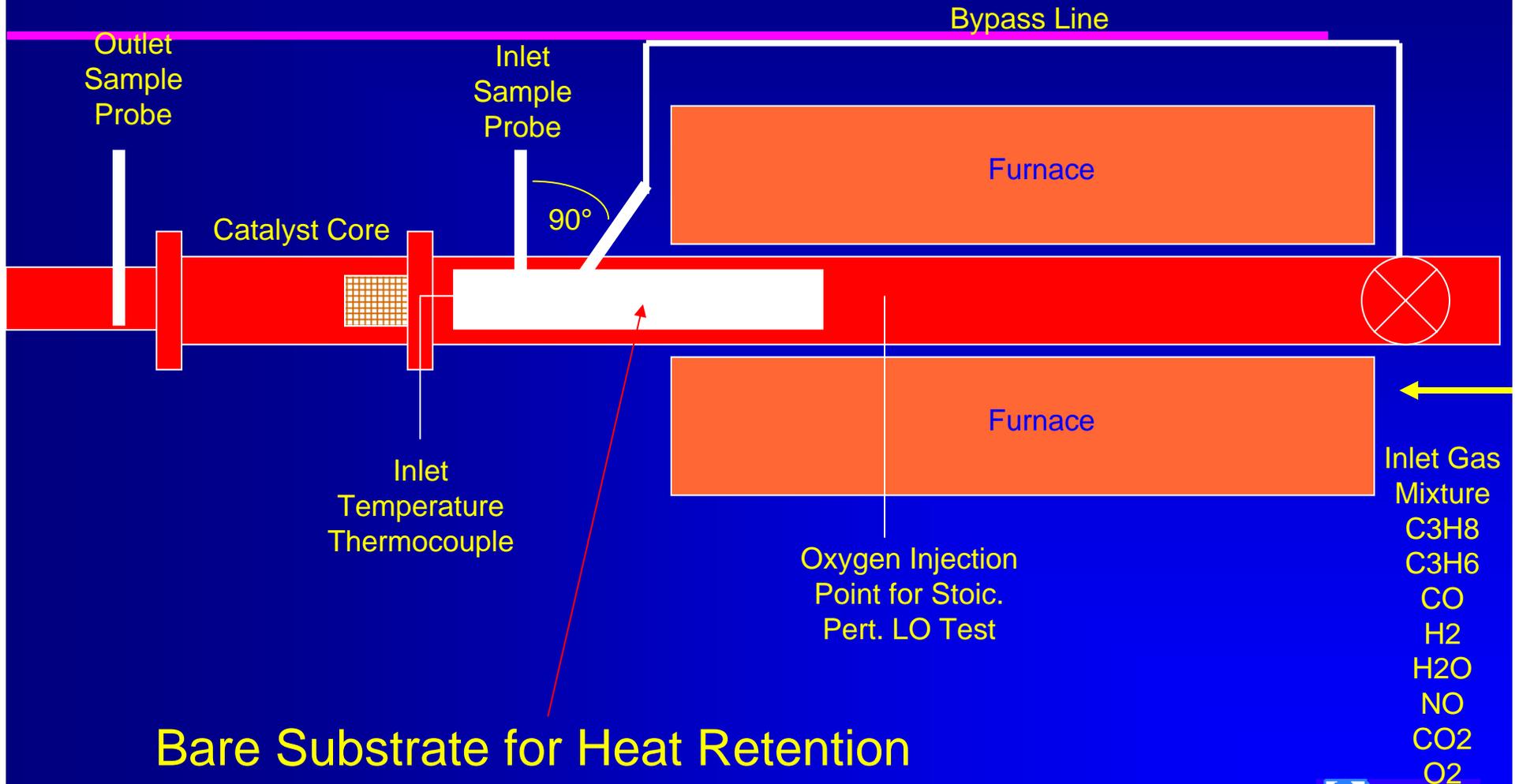
Gordon Bartley



# SYNTHETIC GAS REACTOR



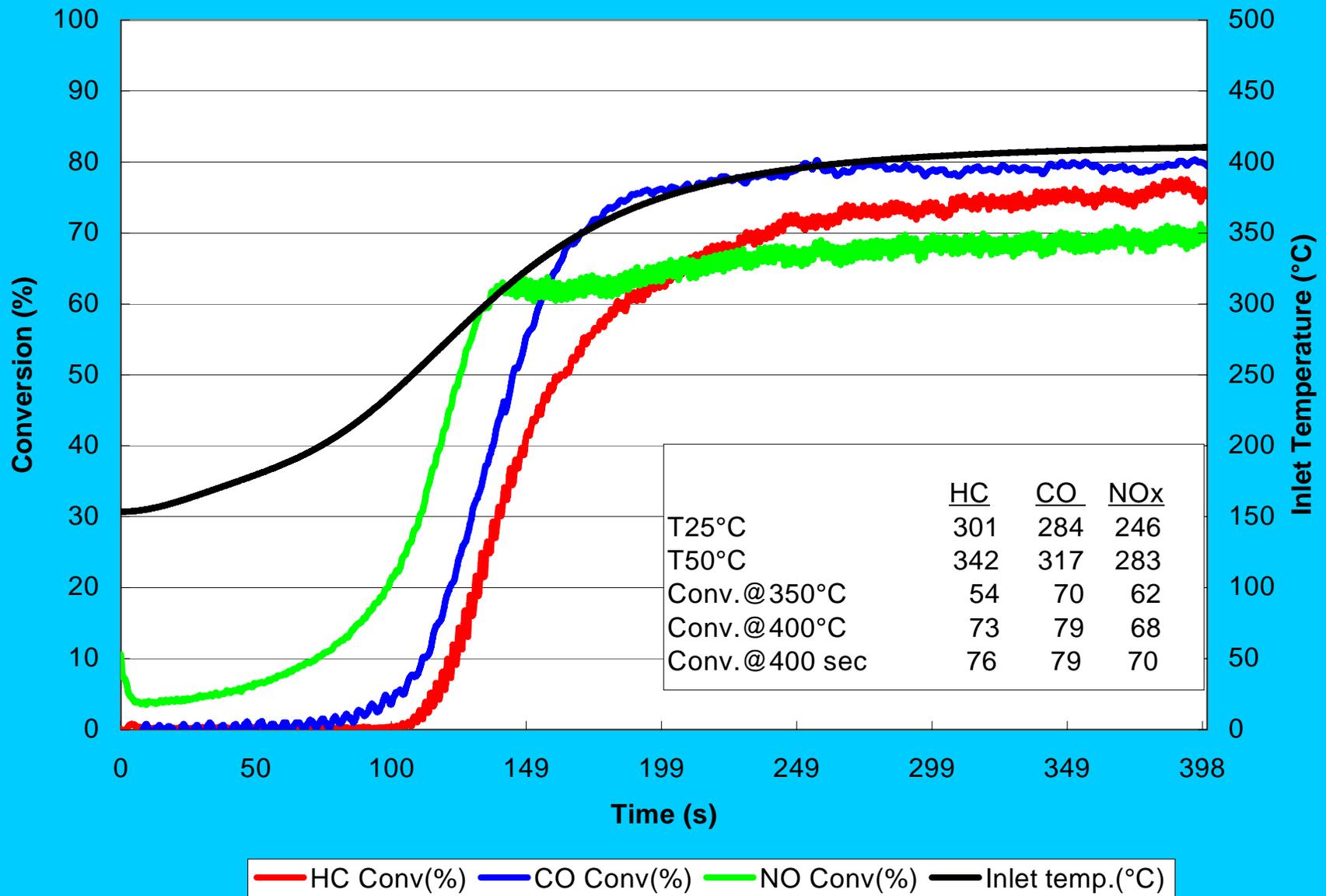
# SGR OPEST LIGHT OFF TEST SETUP - SYSTEM SCHEMATIC



Bare Substrate for Heat Retention  
Modified to Improve Test Characteristics



# SGR SAMPLE RESULTS



Light-Off Test (Perturbation) - LP Core

# OPEST II Test Procedure Outline

---

- ◆ Precondition Catalyst for 20 hours
  - No oil injection
- ◆ (4) SGR tests on 1 inch diameter core
- ◆ 200 hours of aging in the FOCAS™ Rig
  - Test oil injection - 6 quarts
- ◆ (4) SGR tests on 1 inch diameter core



# Program Step - Preconditioned Additional Catalysts

---

- ◆ Reason - Generate enhanced SGR data on preconditioned catalysts
- ◆ Four additional catalysts were preconditioned
  - Catalyst 6, 17, 23, and 27
- ◆ A 1 inch core from each catalyst was removed for enhanced SGR testing



# Program Step - Enhanced SGR Testing

---

- ◆ Core from each 200-hour aged catalyst
  - Tested 4 times
- ◆ Each additional preconditioned core
  - Tested 2 times



# Additional Preconditioned Catalysts Statistics - Enhanced SGR Results

---

- ◆ The average  $T_{50}$  CO for catalyst 27
  - Significantly different than 6, 17, and 23 at the 95% confidence level.
    - » P-value = .0035
- ◆ For the purposes of comparing preconditioned results to the 200-hr aging results, catalyst 27 was excluded.



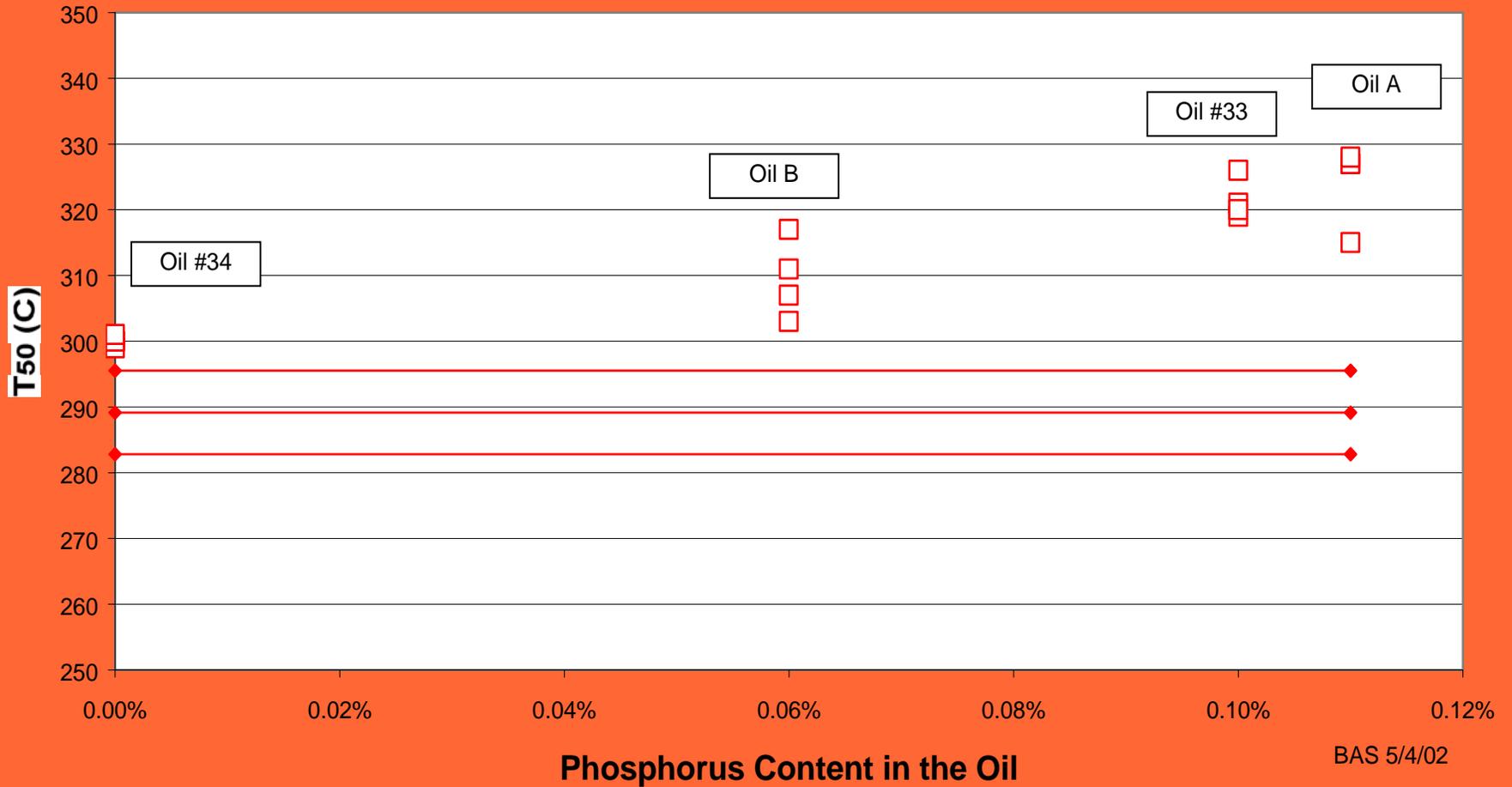
# Presentation of Results and Statistics

---

- ◆ Compare field oil pairs (A and B, 33 and 34)
  - $T_{50}$  HC, CO, and NOx
    - » Scatter plot of data
    - » Statistical analysis of results
      - ◆ Comparisons at 95% confidence level
  - Conversion efficiency at 350° C
    - » Scatter plot of data
    - » Statistical analysis of results
      - ◆ Comparisons at 95% confidence level



# OPEST II - HC - 200 Hrs. Aging Synthetic Gas Reactor Enhanced Stoichiometric Perturbated Procedure



BAS 5/4/02

□ 200 Hours Aging HC    ◆ Precondition HC (Avg-1S)    ◆ Precondition HC (Avg)    ◆ Precondition HC (Avg+1S)

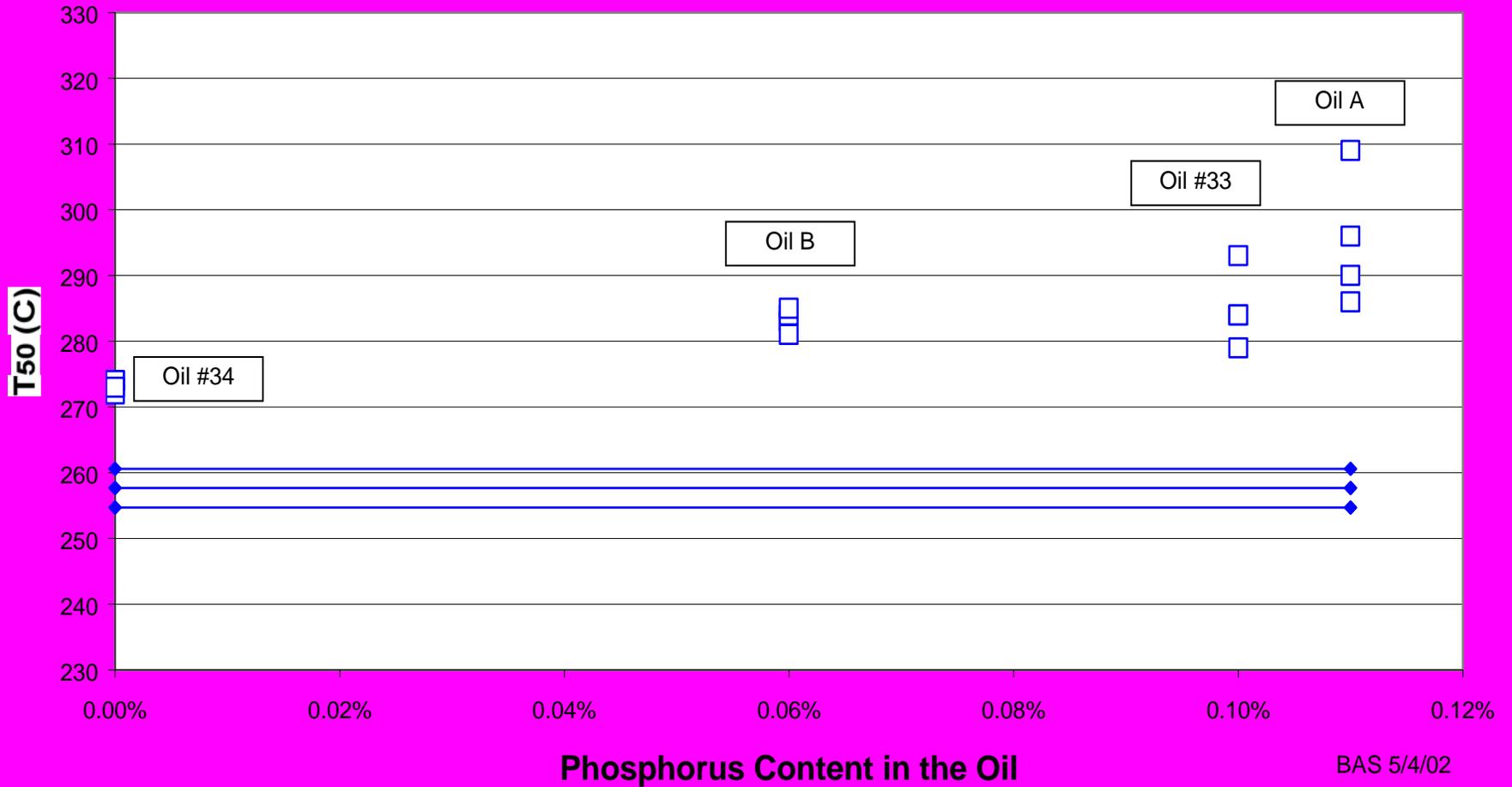
# T<sub>50</sub> HC Statistics

---

- ◆ Oil A > Oil B > No Oil (preconditioned)
- ◆ Oil 33 > Oil 34 > No Oil



# OPEST II - CO - 200 Hrs. Aging Synthetic Gas Reactor Enhanced Stoichiometric Perturbated Procedure



□ 200 Hours Aging CO ◆ Precondition CO (Avg-1S) ◆ Precondition CO (Avg) ◆ Precondition CO (Avg+1S)

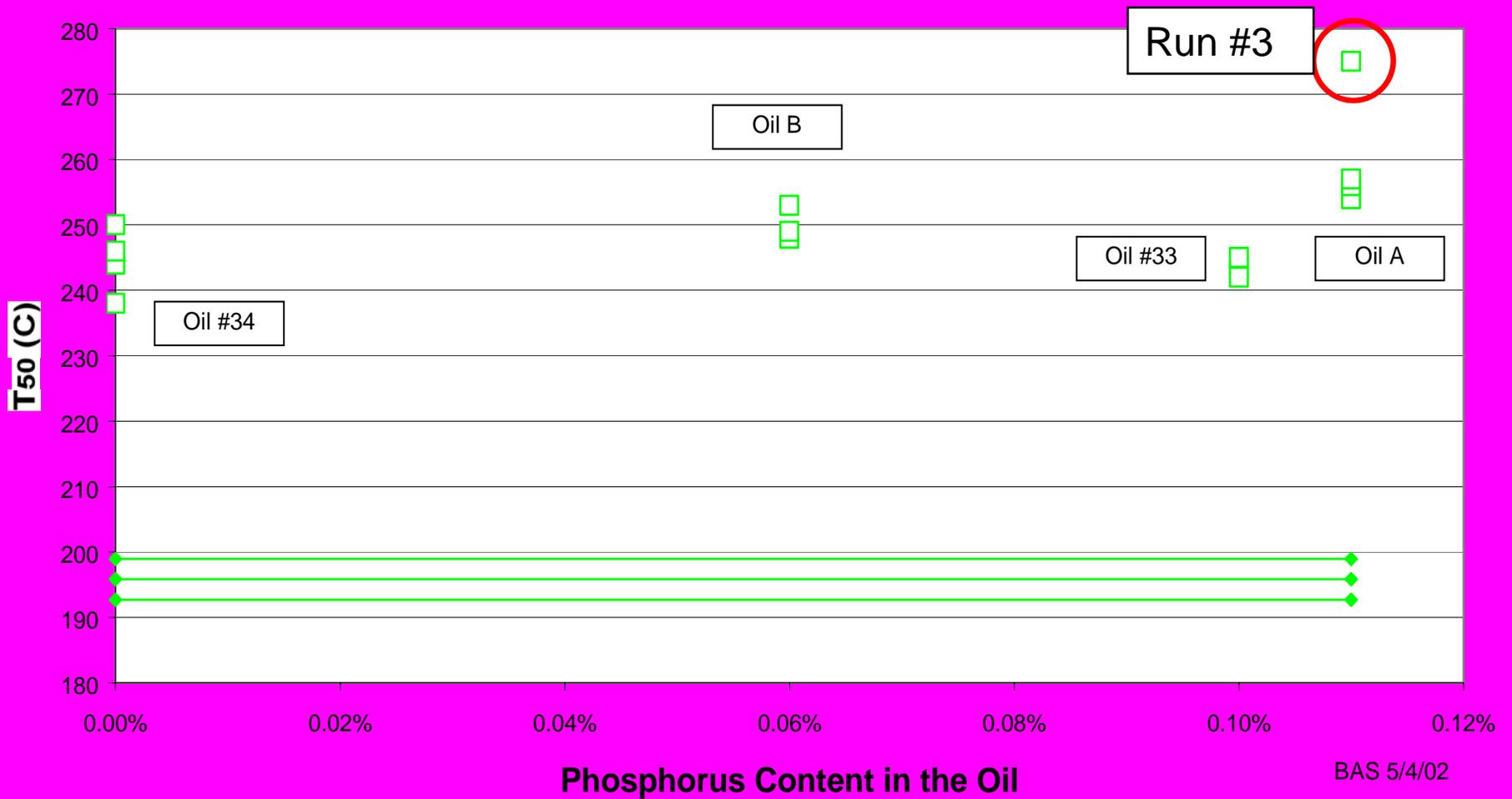
# T<sub>50</sub> CO Statistics

---

- ◆ Oil A > Oil B > No Oil (preconditioned)
- ◆ Oil 33 > Oil 34 > No Oil



# OPEST II - NOx - 200 Hrs. Aging Synthetic Gas Reactor Enhanced Stoichiometric Perturbated Procedure



□ 200 Hours Aging NOx   
◆ Precondition NOx (Avg-1S)   
◆ Precondition NOx (Avg)   
◆ Precondition NOx (avg+1S)

BAS 5/4/02

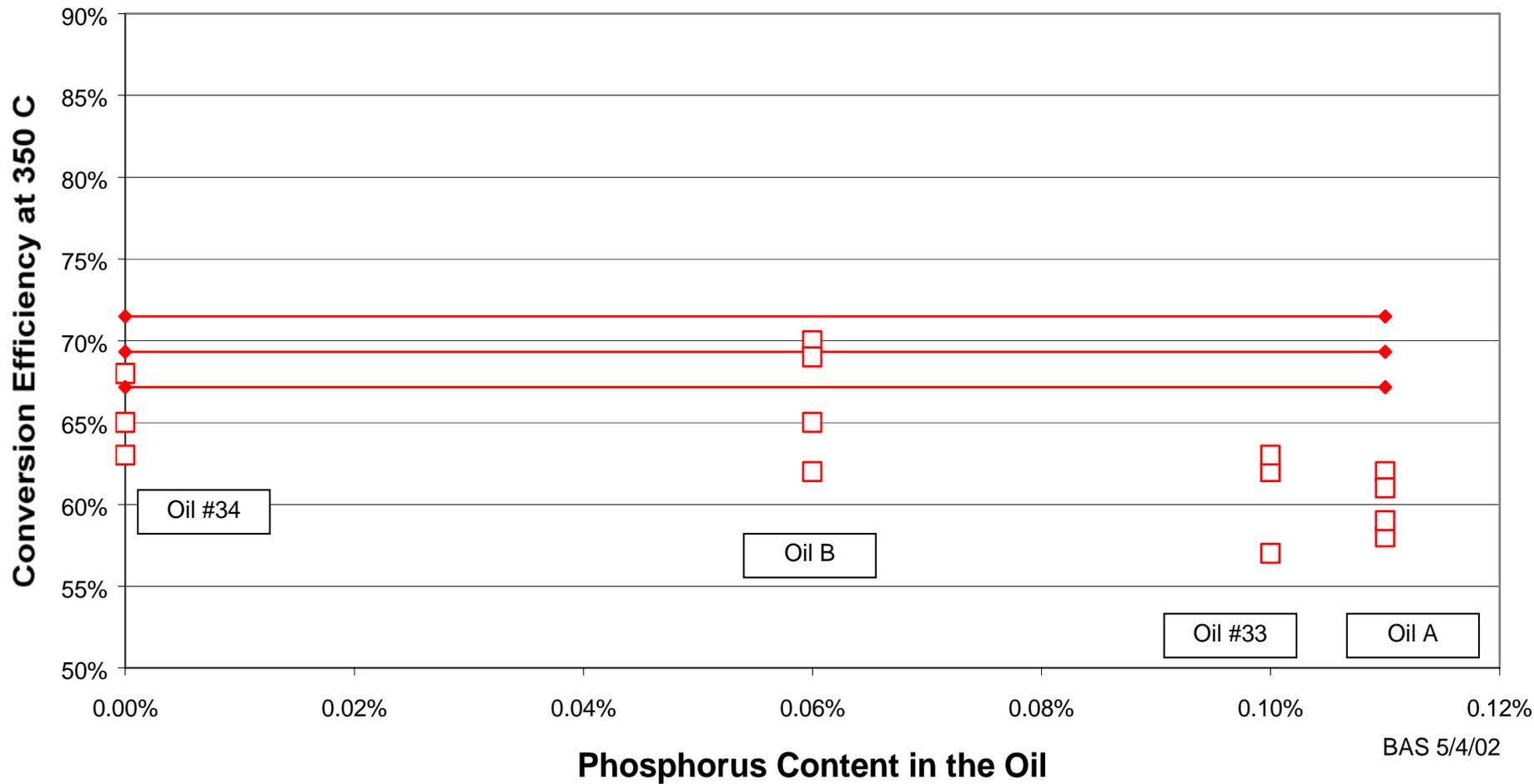
# T<sub>50</sub> NOx Statistics

---

- ◆ With Oil A Run 3
  - Oil A > Oil B > No Oil
- ◆ Without Oil A Run 3
  - Oil A > No Oil, Oil B > No Oil
  - Oil A = Oil B
- ◆ Oil 33 = Oil 34
- ◆ Oil 33 > No Oil
- ◆ Oil 34 > No Oil



# OPEST II - HC - 200 Hrs. Aging Synthetic Gas Reactor Enhanced Stoichiometric Perturbated Procedure



BAS 5/4/02

200 Hours Aging HC
 ◆ Precondition HC (Avg-1S)
 ◆ Precondition HC (Avg)
 ◆ Precondition HC (Avg+1S)

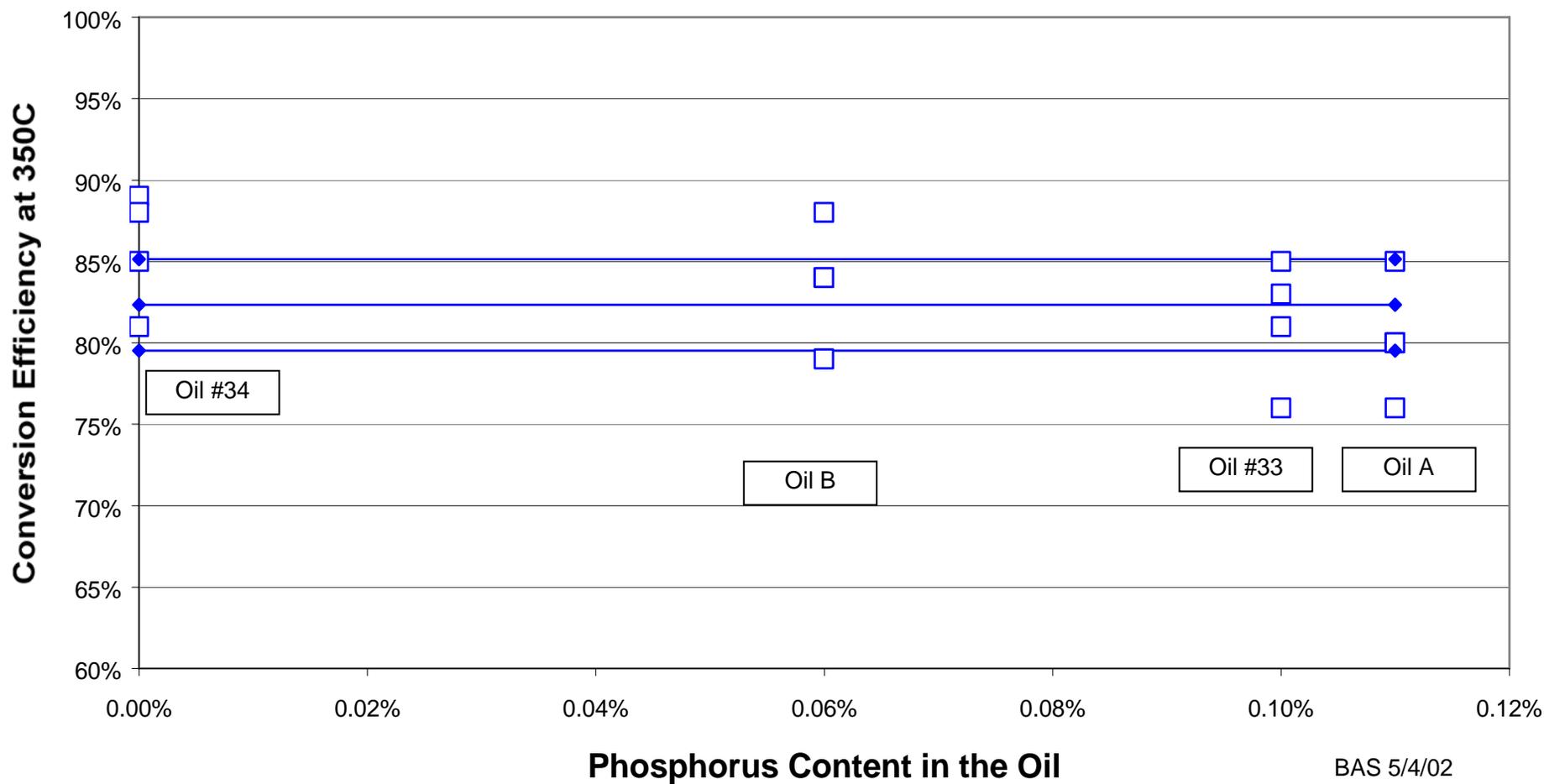
# HC Conversion Efficiency at 350° C

---

- ◆ Oil A < Oil B
- ◆ Oil A < No Oil
- ◆ Oil B = No Oil
- ◆ Oil 33 = Oil 34
- ◆ Oil 33 < Oil 34
- ◆ Oil 34 = No Oil



# OPEST II - CO - 200 Hrs. Aging Synthetic Gas Reactor Enhanced Stoichiometric Perturbated Procedure



□ 200 Hours Aging CO
◆ Precondition CO (Avg-1S)
◆ Precondition CO (Avg)
◆ Precondition CO (Avg+1S)

BAS 5/4/02

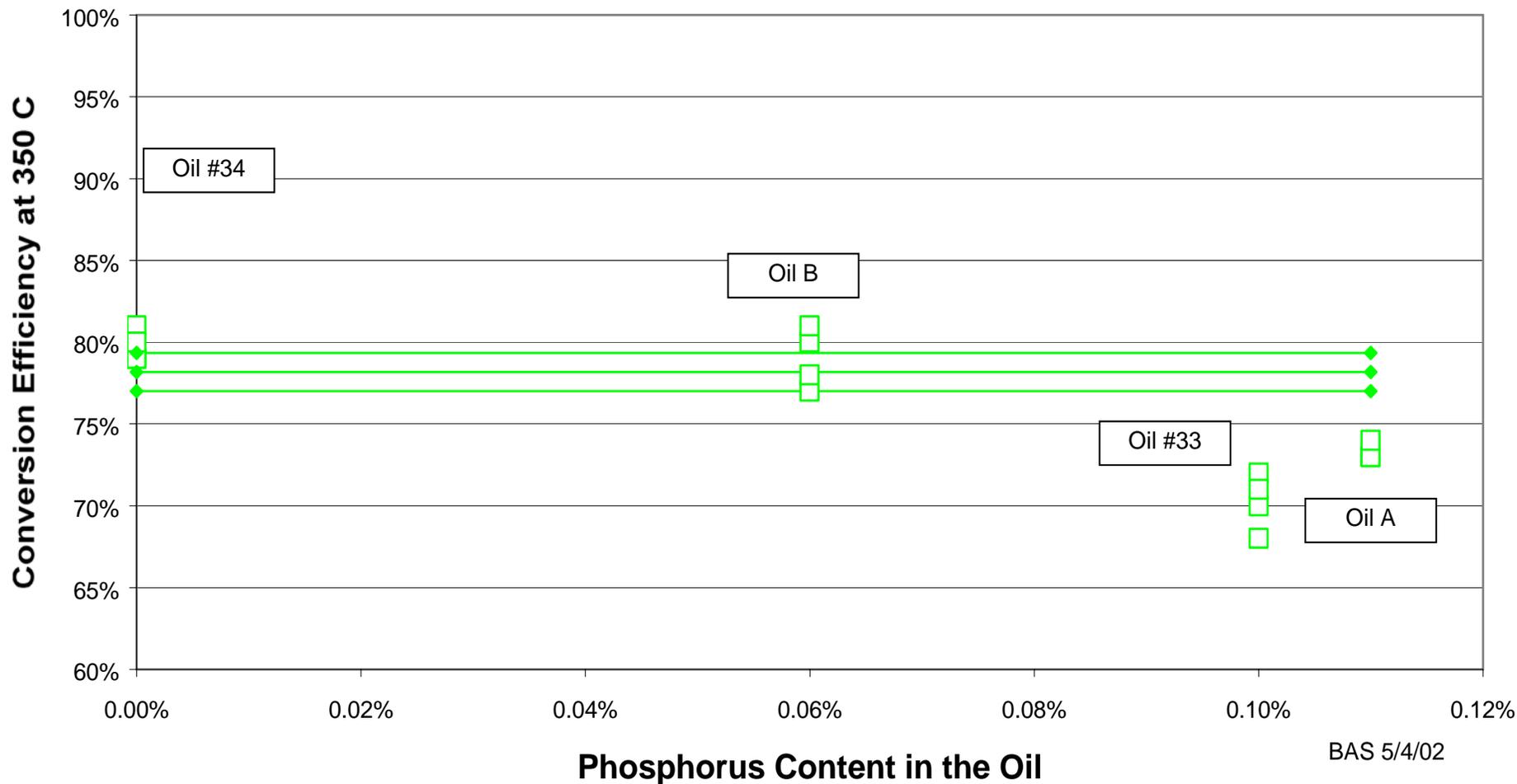
# CO Conversion Efficiency at 350° C

---

- ◆ No significant difference



# OPEST II - NOX - 200 Hrs. Aging Synthetic Gas Reactor Enhanced Stoichiometric Perturbated Procedure



BAS 5/4/02

200 Hours Aging NOx
 ◆ Precondition NOx (Avg-1S)
 ◆ Precondition NOx (Avg)
 ◆ Precondition NOx (Avg+1S)

# NO<sub>x</sub> Conversion Efficiency at 350° C

---

- ◆ Oil A < Oil B
- ◆ Oil A < No Oil
- ◆ Oil B = No Oil
- ◆ Oil 33 < Oil 34
- ◆ Oil 33 < No Oil
- ◆ Oil 34 = No Oil



# Conclusion

---

- ◆ Four catalyst cores were each aged for 200 hours in the FOCAS™ Rig with a test oil.
- ◆ Four test oils - two field tested pairs
  - A and B / Oil 33 and Oil 34
- ◆ With regards to SGR tests on cores from the 200-hour aged catalysts, statistical differences exist between Oil A and Oil B and also between Oil 33 and Oil 34 at the 95% confidence level.



# OPEST II Task Force Concerns

---

- ◆ FOCAS is not a fired engine
  - Other variables?
- ◆ Phosphorus volatility was found to be an important variable
  - First report at the May 2002 meeting
- ◆ Not enough time to be included in GF-4
- ◆ Some concern about discrimination scale



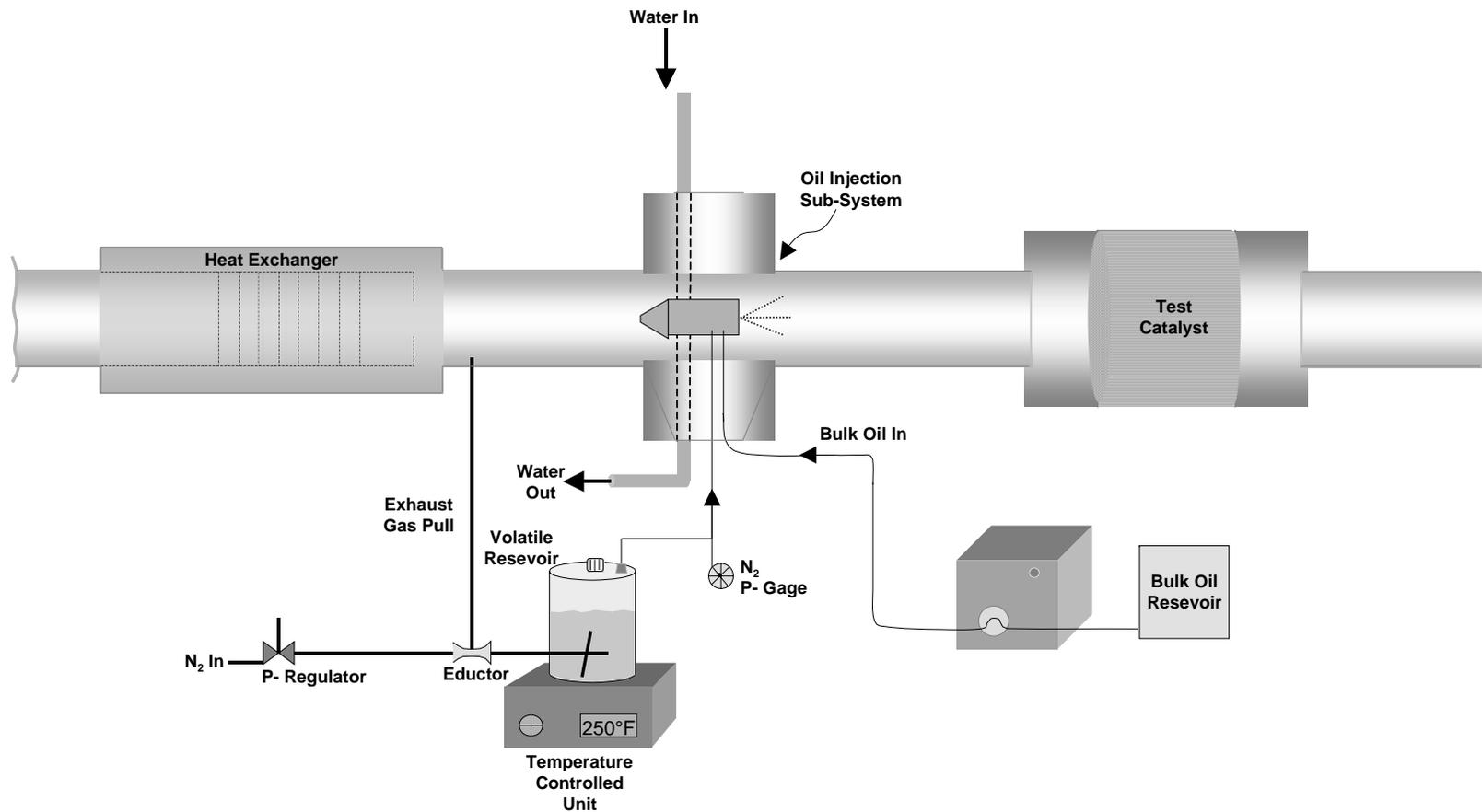
# Update

---

- ◆ Full report of OPEST II was written and distributed
- ◆ FOCAS
  - ◆ Units are currently used for catalyst aging
    - Alternative to engines
    - With and without oil injection
- ◆ SwRI has applied for a patent to add volatile phosphorus from the oil to the FOCAS aging cycle.



# Schematic of the proposed volatilization sub-system Integrated into the FOCAS Rig



The approach proposed for volatilized oil consumption utilizes the pressurized nitrogen system to add in volatilized fractions of P from the oil, through the currently existing oil injection valve.

**Attachment No. 7**

**GF-5 Emissions System  
Compatibility Improvement Team**

**Report to ILSAC/Oil  
6/29/05**

# GF-5 Emissions Team Charter

- The charter of the GF-5 Emissions System Compatibility Improvement Team is to evaluate potential methods for determining the impact of GF-5 engine oil formulations on emission system function and durability. The focus will be the impact of phosphorus and sulfur on catalysts and oxygen sensors. The Team will consider physical, bench, field, and engine tests as an alternative to elemental limits. The Team will make a recommendation to the GF-5 ILSAC/Oil committee by 1/1/2007.

# Emission Team Meeting

- First Meeting held 6/28/05.
- 16 members and guests in attendance.
- Presentation by Ted Selby on engine oil phosphorus and sulfur volatility.
- Presentation by Brent Schoffner on OPEST II.
- Presentations will be attached to the Emissions Team meeting minutes and distributed to ILSAC/Oil.
- Action items and questions compiled

# Action Items

- Develop a set of reference oils for correlation work between field testing and bench, physical, chemical, and dyno testing.
- Compile a bibliography of relevant technical papers.
- Create a spread sheet of field testing either completed or running for emissions system durability. The spread sheet to include lube description, engine type, duty cycle, catalyst and oxygen sensor description, engine durability data, emission system durability data, bench test data and when the data will be available for presentation to the Team.
- Spread sheet to be sent out for comments on parameters with return in 3 weeks and data completion 3 weeks later.

# Questions for ILSAC/Oil

- Will elemental limits be dropped if an acceptable test is developed?
- Is there support from ILSAC/Oil members for an alternative test to elemental limits?
- What is the technical justification for a sulfur limit?
- Is data generated on current systems relevant to 2009?
- Is it a reasonable assumption that if phosphorus exits the engine it is deposited on the catalyst? What is the phosphorus capture efficiency of the catalyst?
- The Team has concerns that we can not meet the 1/1/2007 dead line to have a recommendation for ILSAC/Oil. Is the 1/1/2007 date appropriate for GF-5 timing? ILSAC/Oil should continue to work on elemental limits for GF-5.

---

---

**SPREADSHEET PARAMETERS FOR LUBE IMPACT ON PC GASOLINE  
EMISSIONS STUDIES**

➤ **Vehicle/Engine Description including Field and Dyno Testing**

- Vehicle Make/Model/Year
- Engine
- Repeat Runs?

➤ **Duty Cycle**

- Drain Internal
- Test Length
- Field Test Description
- Dyno Test Description
- Oil Consumption

➤ **Emission System Description**

- Oxygen Sensor
- Light off Catalyst – Size/Metal Loading/Support
- Under Floor Catalyst –Size/Metal Loading/Support

➤ **Engine Durability Data**

- Value Train Wear Measurements
- Used Engine Oil Analysis
- Engine Deposit Rating

➤ **E Missions System Durability Data**

- Used CATALYST light off time and temperature compared to new catalyst
- Used CATALYST analysis for lube related elements
- Mass balance of new oil, used oil and used catalyst.

➤ **Lubricant Description**

- **Fuel Description**
  - Test Fuel
  - Catalyst Analysis Fuel
  
- **Related Data on Lubricant**
  - Physical Analysis
  - Chemical Analysis
  - Bench Tests
  - Sequence Tests

## **Conclusions of Lube Impact on Emission System Durability**